Each employee of AMA Analytical Services is issued a numbered "Employee Handbook" when hired. This handbook addresses general policies of the Aerosol Monitoring & Analysis Companies. All employees must abide by these policies. Failure to do so may result in disciplinary action up to and including dismissal from the firm.

Outlined below are specific policies and philosophies of AMA Analytical Services. Adherence to these policies by both management and laboratory staff is mandatory.

2.1 Quality

AMA Analytical Services is committed to providing the highest quality and most accurate results as practical for the laboratory tests performed. Analytical quality and reproducibility occur by:

- Developing high standards
- Understanding and following standard operating procedures
- Using properly calibrated equipment and removing from service all non-compliant equipment.
- Using properly trained analysts.
- Following quality control procedures.

It is the policy of AMA Analytical Services, Inc. to maintain the highest degree of excellence and proficiency to produce the best analytical results possible, consistent with statutory and regulatory requirements, and customer needs. To achieve this management is committed to the development and implementation of the management system and continually improving its effectiveness. Additionally, it is the duty of top management to communicate the importance of meeting customer requirements as well as statutory and regulatory requirements to the rest of the organization.

AMA Analytical Services, Inc. is committed to following all policies of AIHA, NYLAP, NY ELAP, NELAC, and ISO 17025. It is the responsibility of the President, Laboratory Director, Laboratory Managers, and the Technical Managers to ensure the policies are followed.

2.2 Ethics & Conflict of Interest

At AMA Analytical Services, the one thing we are focused on is getting results accurate and reliable with unprecedented customer service. Simply stated, we get it right. AMA separates itself from its competitors by focusing on providing truthful and accurate information, advice or counseling to our clients. This policy applies to every officer, manager, and employee, regardless of job title or job function. It defines the way in which we conduct ourselves in the performance of our work, be it administrative, professional or technical.

It is the belief of the AMA Companies that all work-related activities be conducted in an ethical manner consistent with the norms of society and free from undue pressure from internal or external sources, which might affect our independence upon providing competent information to our clients. To ensure that all managers and employees fully understand and commit to the importance of data integrity and ethics, we have developed a Data Integrity Plan & Ethics Agreement (see SOP 1002).

Each person will avoid circumstances where a compromise of professional judgment or conflict of interest may arise. (Detailed examples of this are provided in the Employee

Handbook and in the "Code of Professional Ethics for Industrial Hygienists". See also section 2.15 of this manual).

If at any time you believe you are asked to perform tasks for which you have not been properly trained or in a manner, which is in conflict with the procedures outlined in this manual, you must bring the matter to the attention of your direct supervisor or the President of the company

2.3 Staff Employment

All technical staff, excepting laboratory technicians (analysts, technical managers, QC managers, and laboratory director) must have a minimum of a Bachelors Degree (B.A. or B.S.) preferably in a science discipline such as geology, mineralogy, chemistry, life sciences, or physics, from an accredited college or university. Technical and QC managers must have at least 3 years prior laboratory experience. Laboratory Technicians must have a diploma from an accredited secondary school (high school). No exceptions to this policy can be made.

2.4 Technical Training

AMA Analytical Services believes strongly that quality is related to proper and effective training. All analysts must be properly trained and qualified before being allowed to perform analyses for clients. Technical training consists of both detailed in-house training and external courses. All training is conducted by senior analysts who have the appropriate education, training, and experience.

In-house training consists of detailed training in the policies of the company and standard operating procedures for the analytical tests, quality control procedures and the use of equipment. An analyst must demonstrate proficiency to the Lab Manager in regard to a specific test, protocol or use of equipment and must be rated as proficient by quality control procedures prior to performing the procedure. The duration of training varies depending of the individual analytes. Factors determining the duration include mastery of necessary skills, initial demonstration of proficiency, and continuing demonstration of proficiency. Details of specific requirements for each training program are found in the specific training outlines.

Examination of continuing demonstrations of proficiency, internal and external proficiency test, routine QC samples, client feedback, and periodic performance reviews will be used by supervisors to identify continuing training needs.

2.4.1 Documentation of Training

Training records are kept on file for all employees who perform technical functions in the laboratory. The following records, at a minimum, shall be kept on file for each technical employee:

1) Record of successful completion of any specialized training courses.
2) Documentation of any in-house training received, showing dates and times of training, name of trainer, and topics or procedures covered.
3) Documentation demonstrating initial proficiency in analytical techniques and procedures performed by the employee.
4) Documentation of specific SOPs that analyst is authorized to perform.
5) Documentation of any additional or continuing training received by the employee.
6) Records documenting continued proficiency in laboratory procedures performed.

Employee training records are reviewed on an annual basis during performance evaluations to ensure that all necessary documentation of their commitment and adherence to the policies and procedures in the quality system is available.

2.5 Safety

AMA Analytical Services is committed to the safety of its employees. Aerosol Monitoring and Analysis, Inc., the affiliated company of AMA Analytical Services, has developed and implemented a mandatory safety program. All employees of the company are issued a numbered copy of the safety program and are responsible for understanding the safety policies and abiding by these policies. AMA Analytical Services has designated a Laboratory Safety Officer. The Laboratory Safety Officer is responsible for developing and implementing additional safety procedures and policies related to laboratory practices. The designated Laboratory Safety officer as part of training briefs all new employees of AMA Analytical Services on the safety policies and procedures.

Numbered duplicates of the "Corporate Safety Program" and "Laboratory Safety Program" are issued to each employee.

2.6 Ergonomics

AMA Analytical Services is dedicated to providing quality services to its clients. To provide the service required in the asbestos industry, the laboratory is committed to analyzing samples 24 hours a day, 365 days per year.

Management is aware that laboratory analyses can be demanding for microscopists and technicians. To ensure quality control and reduce fatigue and stress of microscopists, management is responsible for monitoring the operation and to take necessary steps to reduce fatigue and stress.

2.7 Laboratory Equipment

All new laboratory equipment is inspected and appropriate checks are performed to determine the suitability and capability of the equipment for analysis. Equipment is not placed in service until its performance has been verified to meet the minimum standards for performance of the analytical methods. Periodic calibrations and checks of performance for analytical equipment in service are also conducted. NIST-traceable calibration materials shall be used to verify performance standards, when possible. Equipment that fails to meet minimum performance or calibration requirements is taken out of service, and is not placed back in service until performance is documented to meet the requirements of the performed method. Only authorized personnel trained in their proper use may operate laboratory equipment. No laboratory equipment is outside the direct control of the laboratory. Instruction manuals, when available, are kept in the same room as the equipment.

2.7.1 Out of Service Equipment

When laboratory equipment is out of service, the following steps are taken:

1. Dismantled to a point where it cannot be used.
2. If portable, removed from the work area.
3. Unplugged and the label "Out of Service, Do Not Use" is attached to the instrument on or near critical controls.

In all situations, analysts qualified to use the machine are informed that the machine is out of service.

If a piece of equipment is sent out for service to an external company, upon its return it is calibrated, when possible, before being placed back into service.

2.8 Reporting of Analytical Results

2.8.1 Electronic Reporting

In accordance with section 2.8.2, client confidentiality, sample results are only electronically transferred to fax numbers and an email address specified on the chain of custody. To insure the integrity of email results, only secure PDF files are transmitted.

2.8.2 Client Confidentiality

Sample results are only reported to the person(s) specified on the chain of custody. If an individual not specified on the chain of custody requests results they are not released until we have written authorization from the person specified on the chain of custody. If special reporting requirements to outside authorities are required by regulation or contract specifications, the client shall provide the laboratory with specific instructions about the project to ensure that all reporting requirements are met. All client records are maintained in a confidential and secure manner. Also see section 5.2.4.

2.8.3 Analytical Discrepancies

If a set of results has been reported to the client and any of the results on the report are found to be in error, through QC or by any other means, the client is informed of the change in results as soon as we are aware of the problem, and no later than 24 hours after discovery of the problem.

When a client questions a result the following steps are used:

1. The report is compared to the original analysis sheets and checked for errors. Quality control analyses and procedures pertaining to the result in question are checked for completeness and errors.
2. The client is notified if this corrects the problem.
3. If the problem is not resolved the sample is analyzed again.
4. The client is notified of the second analysis.
5. If the problem is still not resolved the client is offered a portion of any remaining sample, which they may send to another laboratory.

After the results for the samples in question are corrected further, investigation is conducted to find the source of the problem and the effect on any other sample results. If the investigation shows that samples must be reanalyzed, the clients are notified and a revised report is issued. If the investigation shows that the discrepancy results from a systemic problem and not a random event, changes are made by the Lab Manager and Lab Director to correct the problem. The necessary work is halted until the problem is resolved to the satisfaction of the Lab Manager and the Laboratory Director.

2.8.4 Revised Reports

Occasionally, if a result or other pertinent information is found to be incorrect on the original report, changes must be made to correct a Certificate of Analysis that has already been shipped to the client. If a revised report must be issued, a revision number and date of revision shall be

included on the new report. This information, along with the original report date, shall be included on the report, clearly indicating that this is a revision of a previously released report. The client shall be informed of all changes that have been made to the original report when the revision is issued.

2.8.5 Significant Digits

Most analytical results generated in the laboratory have two significant digits. Some, but not all of the database modules have the capacity to reduce reported results to proper significant digits. Due to limitations of some of the database modules used to generate reports, results are printed with two decimal places. A footnote on each analytical report is used to indicate the number of significant figures appropriate for the reported results.

2.8.6 Sampling Material and Procedures

All samples must be submitted in sealed packaging. Zip-lock backs or whirl packs are acceptable, especially for bulk asbestos, lead paint, and lead soil samples. Lead wipes may be submitted in bags, however, it is preferred that they are submitted in 50mL centrifuge tubes. Lead in air samples shall be submitted on 37mm cassettes with 0.8-micron porosity MCE filters. PCM Air samples shall be submitted on 0.45 - 1.2-micron porosity MCE filters. TEM Air samples shall be submitted on 0.45 – 1.2-micron porosity MCE filters, depending on the type of analysis that is requested. Samples for mold spores shall be submitted using a spore trap cassette or a collapsed MCE filter slide from an inertial impact sampler. None of these samples require preservatives. Lead in water samples may be submitted in 250mL – 1L bottles, depending upon the sampling guidelines being followed. They need to be preserved with nitric acid (HNO_3). This can be done in the field, or can be performed upon receipt of the samples at the laboratory. All sampling media is available to client upon request. Additional information regarding the appropriate sampling media can be found in SOP 101 of Appendix A. Samples can be received by the laboratory via US Mail, overnight courier, courier, and in-person drop off. No special precautions need to be taken except to make sure that all containers are sealed so that the sample integrity is maintained in shipping.

2.9 Deviations from Standard Procedures / Non-Conforming Work

Client Requested

Deviation from the procedures in this guideline or any Standard Operating Procedure used by AMA may be taken under the following circumstances:

1. The client specifically requests that a different procedure be followed.
2. Sample receipt condition, sample quantity, or other conditions prevent adherence to written procedures.

The client and the technical manager or lab director must agree upon and approve any deviation from written procedures after discussion; otherwise the analysis shall be terminated. The deviations to written procedures must be documented in the job file and on the sample report.

Internal

When non-conforming work is discovered, it will immediately be investigated by the laboratory director, technical manager, or the most senior analyst present. Any one of these people alone and/or in conjunction with the laboratory director has the authority and responsibility to stop work and conduct an investigation as necessary. The investigation will include and document:

- The nature of the non-conformity
- The seriousness of the non-conformity
- The effect on analytical results
- Steps needed to correct the non-conformity.

Depending on the outcome of the investigation all or some of the following actions may be taken:

- Suspend the analysis
- Contact the client
- Begin a corrective action investigation
- Restart the analysis
- Retrain the employee
- Fix or decommission any machinery responsible

The investigation will be documented in the database by the assigned investigator. When the investigation, any corrective actions, and follow up are complete the procedure will be allowed to continue upon the authorization and approval of the technical manager and/or the laboratory director.

2.10 Outside Support and Supplies

Only quality products and reagents are used at AMA Analytical Services, Inc. All vendors and their products are checked to ensure overall quality and suitability for their intended laboratory use. All products and services shall be traceable to NIST whenever possible as described in section 2.18 of this manual. Reagents and standards are assigned an expiration date or re-verification date when received. All shipments are checked at time of receipt for damage, completeness, and correctness of order. Appendix E is a list of all of vendors used by AMA Analytical Services, Inc. Any performance verification or contamination checks that are required on reagents or supplies to ensure suitability of the lot are performed prior to use in the laboratory, and also at assigned re-verification dates. Chemicals and reagents are stored in appropriate safety cabinets, and all materials are stored in such a manner as to prevent contamination or damage to the supplies. The supply officer is responsible for ordering, verification, and checking all supplies

Most analytical equipment at AMA Analytical Services, Inc. is redundant; therefore subcontractors are rarely used for analysis. However, when the need for subcontracting work arises, the subcontracted laboratory must carry the necessary accreditations and be rated as proficient by the applicable accrediting authority for the analysis in question.
When analysis must be subcontracted, the firms used must be NVLAP, AIHA or ELLAP accredited depending on the need of the client.

2.10.1 Evaluation of Suppliers

AMA will evaluate suppliers of critical consumables, supplies, and services that affect the quality of testing and calibration and will maintain records of these evaluations, listing those that are approved. A competent reference material supplier is an NMI or an accredited reference material provider (RMP) that conforms with ISO Guide 34 in combination with ISO/IEC 17025 and is traceable to NIST. Conformance is demonstrated through accreditation by an ILAC recognized signatory.

Suppliers are also evaluated based the following criteria.

- Reputation of supplier
- Correctness of Orders
- Availability of desired products
- Certification / Traceability of products and/or services when available
- Do products meet promised specifications?
- Customer Service

The supply officer is responsible for evaluating suppliers. Documentation of supplier evaluations is kept with the list of suppliers and documented in the monthly QA/QC meeting

2.10.1.1 Purchasing of Supplies and Services

Every week each department takes inventory of supplies on hand and makes a list of all supplies needed. The supply officer places orders, with vendors from the approved list, based on availability, prices, and shipping costs of the supplies. If an approved vendor does not have the desired product, research is conducted to find a reputable vendor who does. If the vendor meets our needs, they are added to the approved list.

2.10.2 Subcontracting of Analytical Work

When analysis must be subcontracted, the firms used must be NVLAP, AIHA or ELLAP, or NY ELAP/NELAP accredited depending on the need of the client. TEM bulk and air and PCM samples are never subcontracted. If a sample must be subcontracted, the client is notified of the arrangement, preferably in writing and, when possible, approval is attained prior to subcontracting, preferably in writing. AMA is responsible to the customer for the subcontractor's work, except in the case where the customer or regulatory authority specifies which subcontractor is to be used.

Typically, it is the responsibility of the Business Development Manager to procure the subcontracted laboratory, to discuss with the client the reason(s) subcontracting may be necessary, to obtain approval for subcontracting, and to maintain the list of approved subcontracted laboratories that meet necessary requirements. The Business Development Manager also ensures that client deadlines are met for subcontracted work and that subcontracted results are reported in a manner that is satisfactory to the client.

AMA utilizes the following laboratories for subcontracted work and keeps updated copies of those laboratories' accreditations for the subcontracted work in question:

ASBESTOS – PLM

Scientific Analytical Institute (SAI) - NVLAP Lab Code: 200664-0~~Carolina Environmental NVLAP #101768-0~~
4604 Dundas Drive
Greensboro, NC 27407
(336) 292-3888
~~107 New Edition Court~~
~~Cary, NC 27511~~
(for non-NY State PLM samples)

LEAD (Pb)

Microbac Laboratories – Gascoyne Division – AIHA #100491
2101 Van Deman Street
Baltimore, MD 21224-6697

(for non-NY State Pb Dust Wipes, Soil/Solids, Paint Chips, Airs, & Potable Water samples)

Microbac Laboratories – ~~Waverly Division~~Cortland – ELAP #~~1025~~10795
3821 Buck Drive~~509 Cayuta Avenue~~
Cortland, NY 13045~~Waverly, NY 14892-1532~~
(for NY State Potable Water Samples)

Galson Laboratories – AIHA #100324, NY ELAP #11626
6601 Kirkville Road
East Syracuse, NY 13057-9672
(for NY State and/or non-NY State Airs, Paint Chips, Dust Wipes, Soil/Solids)

Environmental Hazards Services – AIHA # , NY ELAP # 11714
7469 Whitepine Road
Richmond, VA 23237
(for NY State and non NY-State Pb Dust Wipes, Paint Chips, Soil/Solids, TCLPs, & non-Potable Water Samples)

MOLD

Fiberquant Analytical Services – AIHA #101593
5025 S. 33rd Street
Phoenix, AZ 85040
(for all non-viable mold matrices)

Aerobiology Laboratory Associates, Inc. – AIHA EMLAP # 102977
43760 Trade Center Place
Suite 100
Dulles, VA 20166
(for all non-viable & viable mold matrices)

2.11 Annual Audit

Each accreditation program is audited annually. Laboratory procedures, data records and quality control records shall be reviewed to ensure compliance with laboratory policies and procedures, and compliance with policies and requirements of analytical accreditation programs in which AMA participates. When possible we will use the same checklists as external auditors to conduct the audit. The Corporate Quality Assurance Officer shall conduct these audits. Follow up to the annual audits are tracked at the monthly QA/QC meetings. (Also see section 2.16 regarding additional audits). Any deficiencies found during an internal audit shall initiate a corrective action response andshall be recorded in the corrective action report form in the AMA database.

The schedule for internal audits is as follows:

AIHA	Spring
NVLAP	Fall
ELAP	Winter

2.11.1 Executive Review

Quarterly meetings with senior management are held. All QA/QC requirements, management system, and business plan performance are discussed for the current period and the upcoming year. The previous year is reviewed. Plans for resolving any

outstanding issues are formulated, including a completion date and the person responsible for completion of the task. Plans for the New Year are discussed including business development, QA/QC, management system, new equipment, workload, personnel issues and customer service.

Minutes are recorded and issued to the senior management.

2.11.2 Monthly QA/QC and Safety Meetings

Once a month, the Corporate QA/QC officer conducts a monthly review/audit of all laboratory QA/QC operations. Our format is a detailed monthly review and report of all laboratory QA/QC data including Safety, General items, TEM, PLM, PCM, Mold, and AA. The meeting addresses the current status of all the programs including proficiency tests, required QC percentages, past month performance with tables and control charts, and corrections of deficiencies from the past month. A summary report of the details of these meetings is maintained, and includes meeting minutes and QA/QC reports. Attached to this report are the QA/QC printouts, which are filed. The goal of the monthly meetings is to serve as continuous and on-going internal audit process to assure the quality and safety of laboratory procedures and data.

2.12 Use of the NVLAP, AIHA, NY ELAP, & PAACB Logos

The NVLAP, AIHA, and NY ELAP logos are used on certificates of analysis and other laboratory documents to indicate our accreditation by the agencies for analyses of limited matrix types by specific methods. Any documents on which theses logos appear also reference our laboratory identification numbers – NVLAP (101143-0), AIHA (100470), and NY ELAP (10920). The logos are not an endorsement or certification of the laboratory by NVLAP, AIHA, and NY ELAP, or any other government agency.

The PAACB logo is used on certificates of analysis when an analyst certified by PAACB conducted the mold spore analysis. PAACB is an accreditation of the individual analyst. The associated identification number is for the analyst and not the lab. The PAACB ID number is found below the signature of the analyst. At this time, the laboratory does not have an analyst certified by PAACB.

AMA is accredited by NVLAP for the following methods:

Asbestos in bulk materials by Polarized Light Microscopy: EPA-600/M4-82-020 "Interim Method for the Determination of Asbestos in Bulk Insulation Samples".

Asbestos in air by Transmission Electron Microscopy: 40CFR Part 763, Subpart E, Appendix A "Interim Transmission Electron Microscopy Analytical Methods"

AMA is accredited by AIHA for the following methods:

Asbestos Air Analysis – Optical Microscopy, PCM NIOSH 7400

Environmental Lead in paint, soil, air, and dust - SW-846 7000B

Direct Microscopy of Mold spores by ASTM Method D7391 for spore traps only.

AMA is accredited by NY ELAP for the following methods:

Copper and Lead in Potable Water – SM 18-19 3111B and SM 18-19 3113B

Asbestos in Potable Water – EPA 100.2

Asbestos in Friable Material – EPA 600/M4/82/020 and Item ELAP 198.1 of NY ELAP Manual
Asbestos in Non-Friable Material – Item 198.6 of NY ELAP Manual (PLM) and Item 198.4 of NY ELAP Manual (TEM)
Lead in Dust Wipes – SW-846 7000B
Lead in Paint – SW-846 7000B

Asbestos in Air – 40 CFR APX A No. III and NIOSH 7402

2.13 Contracts

All formal bid contracts and subcontract agreements are reviewed by officers of the company. Before a determination is made to enter into or bid on the proposed technical contract, personnel within the company are consulted to determine where technical clarification is needed, and to determine if the laboratory has the resources to meet the specifications of the contract.

2.13.1 Chains of Custody

All chains of custody (COCs) are considered contracts, and as contracts, they are considered legal documents. Therefore, the COC is a legal instrument that documents the receipt, processing, analysis and control of the samples and associated documentation. Since any sample received by the laboratory could be the subject of a lawsuit or be used for evidentiary purposes, the COC is considered the primary document that initiates a process that must be followed for evidence to be legally defensible (acceptable to courts and government agencies). If samples are received where the client indicates on the COC that the samples could be used for evidentiary purposes, the laboratory's Client Services Manager will have a conversation with the client on the particular requirements of the sample set including whether the samples should be returned to the client following analysis or the length of time the samples should be retained by the laboratory. (See section 5 Sample and Document Management.) After a sample has been logged-in, it remains at all times under the control of AMA Analytical Services unless it has been sub-contracted to another laboratory for analysis. If samples need to be sent to another laboratory for any reason, the Client Services Manager will have a conversation with the client to discuss the matter and obtain approval in accordance with our policy and procedures as specified in section 2.10.2 Subcontracting Analytical Services.

Upon receipt and prior to signing a COC, it is determined if the analysis can be completed by the date and time requested and also by the analytical method requested. If it is determined that the client's needs can be met, the COC is signed and processed. If AMA is not able meet the client's requirements as specified on the COC, a senior manager contacts the customer to discuss possible solutions. The conversation with the customer is documented in the appropriate area of the COC. If the issues cannot be resolved, the samples are either returned to the customer or subcontracted to another laboratory per client request.

2.14 Changes in Analytical Methods

When a new analytical method or an updated version of an existing method is issued, all analysts are issued a copy of the method. When necessary, training is given. Before performing the method, all analysts must demonstrate the necessary capability to the satisfaction of the

technical manager. The technical manager documents and signs-off when the analyst has demonstrated proficiency.

2.15 Data Integrity & Ethics Training and Employee Handbook

AMA Analytical Services, Inc. is dedicated to maintaining the highest ethical standards. The company code of ethics is issued to each employee of the AMA companies. Many of the topics outlined in the code of ethics are also covered in the employee handbook. Each employee is issued a numbered handbook. Each employee must sign a receipt for the handbook and acknowledge that they have read the handbook.

To ensure that all managers and employees fully understand and commit to the importance of data integrity and ethics, we have developed a Data Integrity Plan & Ethics Agreement (see SOP 1002). This SOP covers all aspects of our program. Senior managers support and provide initial data integrity training and on-going annual training to laboratory managers and staff. Senior managers ensure that only staff who sign the ethics agreement are allowed to work in the laboratory. The Quality Assurance Manager performs an in-depth review of laboratory reports and the data used to support them on a quarterly basis. The consequences to an employee found to be in violation of the data integrity plan may result in immediate termination, debarment, and/or civil/criminal prosecution.

2.16 Cause Analysis of Problems and Potential Problems

The cause of problems and identification of potential problems is identified by the following methods. Monthly quality control meetings are held at which time problems and solutions are identified. Indications of potential problems are also analyzed during the monthly quality control meetings. Problems and solutions are also identified and discussed at our management review meetings, as are suitability of policies and procedures and customer feedback. Minutes from the QA/QC meetings are kept in the monthly summary books. Minutes from the senior management meetings are kept on file.

The corporate quality assurance officer conducts internal audits annually. These audits serve as a check to see if we are in compliance with QC requirements and also check for potential problems not specifically identified in the QC requirements. In the event that there appears that there is a non-conformance with laboratory procedures and policies, or accreditation program requirements an additional audit will be conducted by the QA/QC officer.

Occasionally AMA Analytical Services, Inc. sends out customer surveys. The data from those surveys is used to help identify and correct any problems not already identified by other methods.

At all times the staff are encouraged to bring to the attention of the management any problems and/or solutions they have. When appropriate staff solutions are used to solve problems.

~~2.16.1~~ 2.16.1 Corrective Action of Problems

When a problem has been identified (i.e., departure from documented policies, procedures, and QC), corrective action is taken which will include ~~analysis~~ an assessment to determine the root cause of the problem. The extent of the corrective action taken depends on the outcome of the root cause investigation. The corrective actions are determined either by the ~~quality control manager, laboratory director, lab~~ technical managers, deputy technical

managers, the quality control manager, the laboratory director, individual analysts, or a consensus of people involved depending on the situation.

The root cause investigation and any corrective measures implemented are documented in the database using the corrective action form (Corrective Action Report). Any corrective actions and/or root cause investigations are discussed at the monthly QA/QC & Safety meeting. When appropriate, the quality control manual and/or the individual SOPs are updated. The staff is issued any changes in policy or procedures.

After action is taken to correct a problem and is documented in the database, follow up examinations are made and documented in the follow-up section of the corrective action form in the database.

The primary individuals responsible for assessing, initiating and/or recommending corrective actions for each type of data is as follows:

TEM: Technical Manager – Andreas Saldivar
PLM: Technical Manager – Peerawut Chaikeenee
PCM: Technical Manager – G. Edward Carney
Metals: Technical Manager – G. Edward Carney
Mold: Technical Manager – Tristan Ward

Each of these individuals is supported by their respective Deputy Technical Manager and technical staff, the QC manager, and the Laboratory Director in a team format. (See section 3.1 for Positions and Responsibilities and section of 3.2 Staffing and Assignments.)

If an analyst determines that the associated QC measurements are unacceptable, he or she will confer with the Technical Manager (or if this person is not available, the Deputy Technical Manager), Laboratory Director, and/or QC Manager. An assessment of all factors will be initiated to determine the root cause of the problem and once the cause(s) of the failure have been found, then corrective actions will be initiated. No results will be released to the Client until satisfactory QC measurements are obtained.

The results of the assessment and the corrective actions will be documented in the appropriate chain of custody and/or log book, and in a Corrective Action Report by the Technical Manager or other manager. Out-of control situations and subsequent corrective actions will also be documented in the monthly QAQC & Safety meeting minutes by the QC Manager. In this format, they will be under formal review by the QC manager. Significant issues will be brought to the attention of senior corporate managers during the quarterly Senior Laboratory Managers Meeting and will be documented in the meeting minutes by the President.

2.16.1.1 Paperwork Errors

When an error is made on a hand written bench sheet or form, each error is crossed out with a single strike through, not erased, and the correct value is entered alongside. All alterations to records are initialed by the person making the correction and dated.

2.16.2 Preventive Action

At all times that problems and potential problems are identified, preventive actions are formulated and discussed as needed, and also at our monthly quality control meeting and quarterly senior management meeting. Ideas and proposed solutions to prevent a problem

from reoccurring or happening in the first place are documented in the minutes of the meeting. Follow up of the solutions are discussed at subsequent meetings.

2.17 Client Complaints

Client satisfaction is a key goal of AMA Analytical Services, Inc. Many client complaints can and are handled through corrective actions. However, most client complaints have no need for corrective action and are generally handled by a manager. Initially, the client services/business development manager, who works with the client to develop a mutually agreeable solution to whatever the issue may be, handles all complaints. If the client services manager is unable to resolve matter, the problem is brought to the attention of the appropriate manager – either to the supervisor of the employee involved or to the manager of the laboratory involved. Together, they work with the client to develop a mutually agreeable solution to whatever the issue may be. In rare instances, client complaints are unable to be resolved by either the client services manager and/or another laboratory manager. These cases are handled by the laboratory director, who works with the client to develop a mutually agreeable solution.

Client complaints are documented in the AMA Analytical Services, Inc. Database and in the senior managers meeting minutes.

2.18 Traceability

Whenever possible, materials, supplies, and certified reference materials procured and used by the laboratory shall be evaluated for the appropriate accreditations e.g., reference materials traceable to NIST through accreditation by an ILAC recognized signatory and accredited to ISO/IEC 17025:2005 and to ISO Guide 34; and external calibration services must be a calibration laboratory that is traceable to NISH and accredited to ISO/IEC 17025:2005 by an ILAC recognized accreditation signatory. This applies to all equipment, supplies, and materials that may affect results generated by the laboratory. If NIST-traceability is not technically possible or reasonable, certified reference materials provided by a competent supplier as defined in section 2.10.1 of this manual, or specified methods and/or consensus standards that are clearly described and agreed to by all parties concerned, shall be used. I. AMA will ensure that purchased supplies, reagents and consumables that affect the quality of tests and/or calibrations are not used until they have been inspected or otherwise verified as complying with standard specifications or requirements as defined in the methods for the tests and/or calibrations concerned. The services and supplies used will comply with the specified requirements, and records of actions taken to check compliance will be maintained.

The supplier of the material shall provide certificates of analysis verifying traceability. Internally prepared calibration materials shall be compared to a traceable certified standard before being used. Any calibrations performed by calibration service providers shall be traceable to to NIST and to ISO/IEC 17025:2005, as evidenced by a certificate of calibration that demonstrates the calibration was performed by a calibration laboratory accredited to this standard Calibration certificates will indicate endorsement by a recognized accreditation body symbol. Certificates will indicate traceability to the SI or reference standard and include the measurement result with the associated uncertainty of measurement.

2.19 Uncertainty of Results

AMA will provide, along with the reported analyte concentration, a calculated estimate of the expanded measurement of uncertainty of any results generated using standard

laboratory test methods in the same units as the analyte concentration when : (1) it is relevant to the validity or application of the test results, or (2) a customer's instructions so require it, or (3) the uncertainty affects compliance to a specification limit . The uncertainty estimates we provide are based on repeated analysis of reference samples. When at least 30 reads of a reference material have been completed, the average percent recovery and standard deviation of all obtained results is calculated. With regard to lead analyses, the uncertainty associated with the lead standard, and the laboratory's sub-sampling of paints and soils will be taken into account. The estimated probable bias of reported test results is reported as a positive or negative in the same units as the analyte, based on the difference between the expected reference sample results and average measured recovery. The estimated precision of the test results is reported as a 95% confidence interval, given as +/- in units of the reported result. This interval is determined as +/- 2 times the standard deviation of the compiled reference sample results. The reported uncertainty is based on at least 30 reads of a reference material of the same matrix or prepared using the same matrix as the associated test results. When reporting measurement uncertainty, the test report will include the coverage factor and confidence level used in the estimations. A coverage factor of approximately 2 is used at the 95% confidence interval. When the test method has a known and uncorrected systematic bias, it will be reported separately from the test result and uncertainty, as a probable bias value.

The following sections provide a specific process to estimate measurement of uncertainty for each type of test performed.

2.19.1 Phase Contrast Microscopy

Definition of the Measurand

- ❑ Fibers on filter

Identification of the Contributors of Uncertainty

- ❑ Scope calibration
- ❑ Sample preparation
- ❑ Analyst calibration

Approaches Used for Estimating Uncertainty

- ❑ Quantified by S_r values derived from repeated analysis of reference samples

2.19.2 Polarized Light Microscopy

Definition of the Measurand

- Asbestos type and quantity

Identification of the Contributors of Uncertainty

- Scope calibration
- Sample preparation
- Analyst calibration

Approaches Used for Estimating Uncertainty

- Quantified by S_r values derived from repeated analysis of reference samples

2.19.3 TEM Air

Not applicable at this time.

2.19.4 TEM NOBs

Not applicable at this time.

2.19.5 TEM Water

Definition of the Measurand

- Asbestos fibers in water

Identification of the Contributors of Uncertainty

- Scope calibration
- Sample preparation
- Variation in grid opening size

Approaches Used for Estimating Uncertainty

- Use of the 95% upper and lower confidence limits based on a Poisson distribution (as discussed in the method)

2.19.6 TEM Settled Dust

Not applicable at this time.

~~2.19.7 Lead Air~~

Definition of the Measurand

- Lead on filter

Identification of the Contributors of Uncertainty

- Measurement of digestate volume
- Variation in instrument performance
- Elution efficiency
- The lead standard

Approaches Used for Estimating Uncertainty

- Repeat analyses of Laboratory Control Samples (LCS)
- Calculate average recovery and SD measurement. Uncertainty is + and – 2 SDs (95% confidence interval)

2.19.8 Lead Water

Definition of the Measurand

- Lead in water

Identification of the Contributors of Uncertainty

- Variation in instrument performance
- The lead standard

Approaches Used for Estimating Uncertainty

- Repeat analyses of Laboratory Control Samples (LCS)
- Calculate average recovery and SD measurement. Uncertainty is + and – 2 SDs (95% confidence interval)

2.19.10 Lead Paint Chips

Definition of the Measurand

- Lead in percent in paint chips

Identification of the Contributors of Uncertainty

- Measurement of sample mass
- Variation in instrument performance
- Elution efficiency
- The lead standard
- Sub-sampling duplicates

Approaches Used for Estimating Uncertainty

- Repeat analyses of Laboratory Control Samples (LCS)
- Calculate average recovery and SD measurement. Uncertainty is + and – 2 SDs (95% confidence interval)

2.19.11 Lead Dust Wipes

Definition of the Measurand

- Lead mass on wipe

Identification of the Contributors of Uncertainty

- Measurement of digestate volume
- Variation in instrument performance
- Elution efficiency
- The lead standard

Approaches Used for Estimating Uncertainty

- Repeat analyses of Laboratory Control Samples (LCS)
- Calculate average recovery and SD measurement. Uncertainty is + and – 2 SDs (95% confidence interval)

2.19.12 Lead Soils

Definition of the Measurand

- Lead in ppm by dry weight in soil

Identification of the Contributors of Uncertainty

- Measurement of sample mass
- Measurement of digestate volume
- Variation in instrument performance
- Elution efficiency
- The lead standard
- Sub-sampling duplicates

Approaches Used for Estimating Uncertainty

- Repeat analyses of Laboratory Control Samples (LCS)
- Calculate average recovery and SD measurement. Uncertainty is + and – 2 SDs (95% confidence interval)

2.19.13 Lead TCLP

Definition of the Measurand

- Lead in ppm by weight in extract

Identification of the Contributors of Uncertainty

- Measurement of digestate volume
- Variation in instrument performance
- Elution efficiency
- The lead standard

Approaches Used for Estimating Uncertainty

- Repeat analyses of Laboratory Control Samples (LCS)
- Calculate average recovery and SD measurement. Uncertainty is + and – 2 SDs (95% confidence interval)

2.19.14 Mold Air

Definition of the Measurand

- Spores on slides

Identification of the Contributors of Uncertainty

- Scope calibration
- Sample preparation
- Analyst calibration

Approaches Used for Estimating Uncertainty

- Quantified by S_r values derived from repeated analysis of reference samples

2.19.15 Mold Tape/Bulk

Definition of the Measurand

- Spores on slides

Identification of the Contributors of Uncertainty

- Scope calibration
- Sample preparation
- Analyst calibration

Approaches Used for Estimating Uncertainty

- Quantified by S_r values derived from repeated analysis of reference samples

2.19.6 Mold Vacuum Dust/Swab

Definition of the Measurand

- Spores on slides

Identification of the Contributors of Uncertainty

- Scope calibration
- Sample preparation
- Analyst calibration

Approaches Used for Estimating Uncertainty

- Quantified by S_r values derived from repeated analysis of reference samples

Outlined in the following subsections are the AMA Analytical Services policies for sample handling and acceptance criteria, documentation and file organization, sample disposition, and sample storage. Specific step-by-step operating procedures for these steps are found in associated SOPs for technical secretaries. Key items and policies are presented in this section.

5.1 Sample Handling and Acceptance Criteria

The Technical Secretary is responsible for sample coordination from the time samples are received until samples are placed in the proper holding area for analysis. Sample coordination responsibilities include:

- Receiving samples
- Accepting or rejecting sample(s) or sample set(s)
- Assigning reference number and establishing a file
- Log-in of samples

5.1.1 Receiving Samples

The Technical Secretary, upon receiving the samples, will check or confirm the following:

- Make sure all forms, especially the chain of custody (COC) forms are properly filled out. If pertinent sampling information, such as date and time sampled is missing, sample layers, etc. is not provided, every effort is made to contact the client and obtain this information. For sample analyses and/or matrices that are affected by method specified holding times that are not able to be determined, AMA will process those samples as though that holding time has been exceeded by either voiding the samples or employing additional preparation techniques as necessary.
- Check shipping package for damage or to see if custody seal is broken.
- Note date and time samples are received by the laboratory.
- Document how samples are shipped to the laboratory (i.e., hand delivered, courier, mail, Federal Express) and condition of package if damaged.
- Confirm that the number of samples submitted matches the number of samples on the client-supplied paperwork.
- Confirm project deadlines for preliminary analytical results and written reports.
- Confirm analytical protocol to be performed, if possible.
- For asbestos water samples: record the temperature of samples at time of receipt on the chain of custody. If the temperature is +/-2° of 4° Celsius or the samples are received more than 48 hours after collection an ozone generator will be required during preparation procedures.
- Confirm that samples have been received in appropriate condition and in appropriate containers as defined in SOP 101 of Appendix A.
- Confirm that samples submitted fall within AMA's accreditation status as specified in SOP 101 of Appendix A.

If there are any questions or concerns regarding the sample(s) or sample set(s), the Technical Secretary will stop and contact the appropriate Laboratory Manager.

5.1.1.1 Unusual Sample Shipments

Before the laboratory will accept samples with unusual conditions or analysis requests, laboratory management must first ascertain our ability to perform the work. The following considerations shall be examined:

1. Does the laboratory have the facilities and equipment to safely handle the sample, through storage, preparation, analysis, and disposal?
2. Does the laboratory have the facilities, equipment, and technical capability to perform the requested analysis on this sample?
3. Can we complete the work within the requested time period.
4. Can we legally perform work on this sample, or would acceptance of the sample create an excessive liability risk for the company?

5.1.1.2 Proficiency Test Samples

As much as possible all proficiency test (PT) samples are handled the same as client samples. When the samples arrive they are logged in and, to the extent possible, put into the regular sample stream. The following procedures are used:

PCM AIHA IHLAP PT , ELAP PT, & Interlab Round Robins

The samples are submitted to the PCM laboratory as regular samples. Samples are logged into the database and submitted to the laboratory in the same manner as any set of client samples. The due date for the initial set of data shall be 5-10 business days. Sample preparation, analysis, and QC are also conducted in the same manner as any set of client samples. Once analysis is completed, the analyst shall enter the data in the AMA database and generate a report as would be done for routine client samples. The report shall be emailed to the Laboratory Director and to the department Technical Manager. The results and data of this initial analysis are submitted to the issuing agency for evaluation. After the initial analysis and QC is complete, the samples are returned the department Technical Manager for distribution to all other PCM analysts who shall be required to complete analysis by the official due date established by the issuing agency. Once all analysts read the samples, the Technical Manager evaluates the data.

TEM NVLAP PT, ELAP PT, & Interlab Round Robins

Sample chain-of-custody and login is handled in the same manner as client samples and are scheduled for analysis by the official due date established by the issuing agency. Once samples have been logged into the AMA database, they are delivered to the department Technical Manager for distribution to all TEM analysts as appropriate. Due to the nature of these samples, it is not possible to handle all PT samples in the exact same manner as client samples. While all analysts participate in the PT rounds, every sample is not read by all the TEM analysts. In some instances, PT rounds are portioned out. Water and air samples are split among the analysts as directed by the department Technical Manager, with each sample being assigned to a single analyst. They are then prepped and analyzed in accordance with the written instructions provided by the issuing agency. TEM unknowns, filters, and bulk samples are prepared in accordance with the written instructions provided by the issuing agency. All analysts read these samples. Where possible and appropriate, analysts enter data into the AMA database and generate a report as would be done for routine client samples, and the report is emailed to the Laboratory Director and department Technical Manager. It is the responsibility of the department Technical Manager to submit data to the issuing agency for evaluation.

Mold AIHA EMLAP Direct Micro Exam, AIHA EMLAP Viable Analysis, & Interlab Round Robins

The direct micro exam is taken by all analysts. This is conducted by having all the mold analysts present at the computer during the 1 hour allotted time period.

Viable samples are handled as described in SOP 909 of Appendix A of this Manual. The due date for viable PT samples shall be 30 calendar days from the date of receipt. Once all analysis and QC has been completed, results shall be entered into the AMA database and a report generated as would be done for routine client samples. The data entered into the AMA database shall be what is submitted to the issuing agency for evaluation. The report shall be emailed to the Laboratory Director and the department Technical Manager. [As of November 2011, AMA Analytical Services, Inc. ceased it participation in the AIHA Viable Analysis accreditation program, and is no longer accredited for these types of samples.]

Round Robin samples are submitted to the mold laboratory as regular samples. Samples are logged into the database and submitted to the laboratory in the same manner as any set of client samples. The due date for the initial set of data shall be 5-10 business days. Sample preparation, analysis, and QC are also conducted in the same manner as any set of client samples. Once analysis is completed, the analyst shall enter the data in the AMA database and generate a report as would be done for routine client samples. The report shall be emailed to the Laboratory Director and department Technical Manager. The results and data of this initial analysis are submitted to the issuing agency for evaluation. After the initial analysis and QC is complete, the samples are returned the department Technical Manager for distribution to all other mold analysts, who shall be required to complete analysis by the official due date established by the issuing agency.

Lead AIHA ELLAP PT, ELAP PT

The samples are submitted to the metals laboratory as regular samples. Samples are logged into the database and submitted to the laboratory in the same manner as any set of client samples. The due date for the initial set of data shall be 5-10 business days. Sample preparation, analysis, and QC are also conducted in the same manner as any set of client samples. Once analysis is completed, the analyst shall enter the data in the AMA database and generate a report as would be done for routine client samples. The report shall be emailed to the Laboratory Director and department Technical Manager. The results and data of this initial analysis are submitted to the issuing agency for evaluation. After the initial analysis and QC is complete, the samples and/or digestates are returned the department Technical Manager for distribution to all other metals analysts, who shall be required, as possible and appropriate, to complete analysis by the official due date established by the issuing agency. Once all analysts complete analysis as possible and appropriate, the Technical Manager evaluates the data.

PLM NVLAP PT, ELAP PT, & Interlab Round Robins

Samples are logged into the database and submitted to the laboratory in the same manner as any set of client samples. The due date for the initial set of data shall be 5-10 business days. Sample preparation, analysis, and QC is also conducted in the same manner as any set of client samples. Once analysis is completed, the analyst

shall enter the data in the AMA database and generate a report as would be done for routine client samples. The report shall be emailed to the Laboratory Director and to the department Technical Manager. The results and data of this initial analysis are submitted to the issuing agency for evaluation. After the initial analysis and QC is complete, the samples are returned the department Technical Manager for distribution to all other PLM analysts who shall be required to complete analysis by the official due date established by the issuing agency. Once all analysts read the samples, the Technical Manager evaluates the data.

5.1.2 Acceptance/Rejection Criteria

Sample(s) or sample set(s) shall be voided/rejected at the time of receipt or log-in under the following conditions:

5.1.2.1 Air Samples and Spore Traps

- If sample cassette has opened during transit.
- The entire sample set is rejected if bulk and air samples are shipped in the same container without being sealed in separate packages.
- Evidence of gross contamination of cassettes or inside of containers.

5.1.2.2 Bulk Samples

- Individual sample to be voided if sample container has ruptured.
- Entire sample set is to be voided if there is evidence of improperly sealed sample containers and visible evidence of debris on outside of individual sample container.
- If multiple samples are provided in the same sealed container rather than individually sealed.

5.1.2.3 Water Samples

- Sample container is ruptured
- Evidence of gross contamination of sample is obvious
- Improper sample container
- Sample is improperly preserved
- Method-specified holding times are exceeded
- Insufficient sample submitted to perform requested analyses

5.2 Documentation and File Organization

5.2.1 Sample Receipt

All sample(s) or sample set(s) are assigned to a unique AMA chain-of-custody (COC) reference number. A unique file folder is created for each sample submittal / Chain of Custody. Any actions taken with the samples, such as rejection of samples and reasons for rejection shall be documented. Any conversations with the client regarding the sample set are documented on the COC form in the space provided for "Client Contact." If the submitted samples are accepted for analysis, then the following steps occur:

- Client and project information are entered in the computer system.
- Unique sample identification numbers are assigned to each sample.
- Verification that each client sample is labeled with the correct laboratory sample ID number as logged into the computer system
- Samples are submitted to the analytical operation for analysis.

5.2.2 Sample Analysis

All original analysis sheets, bench notes, and QC analyses for the sample set are kept in the job file. Any deviations from standard procedures and any conversations with the client regarding the samples shall be documented by the analyst and placed in the job file. Final reports and invoices generated are also kept in the job file. The date and time of analysis, and the date and time results are reported to the client is documented on the COC form and kept in the job file.

5.2.3 Filing ~~Archiving~~ of Documents and Records

After mailing of final reports and invoices is completed, the job file, including a copy of the final report, is scanned into the PCI .pdf file conversion database by the Technical Secretary. The job file containing all of the above mentioned sample data, as well as any documentation submitted by the client with the sample set is then converted to a .pdf file, and stored for ready use in electronic format for a period of at least 12 years. We currently use the Advanced ArchiveIT Enterprise database.

5.2.3.1 Scanning and Indexing Procedures

1. All external paperwork, which consists of the invoice, certificate of analysis, and any supporting documentation that needs to be returned to the client are scanned into the External folder, and a .tif file is created.

2 All associated internal paperwork, which consists of raw data sheets, and any other documentation that is not returned to client is scanned into the Internal folder, and a .tif file is created.

3 The Technical Secretary opens the PCI software and indexes each .tif file by assigning it to the appropriate Chain of Custody number, thereby creating a .pdf file, which is stored as a web document. The file can then be recalled by opening the AMA Search Web Page and typing in the desired Chain of Custody number.

5.2.3.2 Scanning and Indexing QC Procedures

After indexing is complete, it is necessary to check the indexed files for accuracy. A list of all indexed files is compared to a list of job folders for each batch of scanned jobs. If a file exists that is not on the list of job folders that file is opened and the actual Chain of Custody number is compared to assigned Chain of Custody number. If the Chain of Custody numbers do not match, the file is deleted, re-scanned and re-indexed. If the numbers do match, the list of job folders is checked, and any necessary corrections are made. Conversely, if a file that appears on the job folders list is not on the indexed files list, the files before and after the missing job are opened and reviewed. If the missing data has been incorporated into the data of another Chain of Custody, then that file is deleted, and both jobs are re-scanned and re-indexed. If the missing data has not

been incorporated into another Chain of Custody, then the missing job is scanned and indexed.

Once the list of indexed files is compared to the list of job folders, and all jobs are accounted for, a 10% random QC check is required. 10% of the files from a batch of scanned jobs are opened at random, and all of the scanned data is reviewed to ensure that all paperwork associated with any given job is available. If a job is found to be incomplete, any missing paperwork is scanned and indexed. If recurring errors are found during this QC check, an investigation is launched to determine the cause, and resolve the problem.

5.2.3.3 Scanning and Indexing Procedures for Internal Lab Paperwork

Internal laboratory paperwork is also stored electronically for ready use. ~~.~~ These items include, but are not limited to, TEM Verified Counts, proficiency results, AA SDG Reports, AA Log Data, and AA Raw Data, internal audits, management reviews, preventive action records, calculated control limits, instrument calibration data, training records, and any other instrument records. These items constitute internal data that is not necessarily associated with any particular job, but is maintained for general QC purposes. The procedures for scanning and indexing these documents are as follows:

1. Scan the item into the appropriate folder.
2. Rename the scanned file to a name that is appropriate. For example, an SDG report shall be renamed as "SDG XXXX."
3. Open the scanned file and compare the name of the file to the contents of the file in order to ensure that the name is appropriate.
4. Once it is determined that all scanned files are available and have been named appropriately, the associated paperwork is shredded and discarded.

Not all laboratory records are stored as electronic documents or records. However, ~~T~~things such as complaint records, corrective actions, non-conforming work, and records and reports changes are documented and tracked in the AMA Analytical Services' database.

Blank laboratory documents, such as bench sheets, are stored as read-only files on the main server. Original paper copies are stored in the Original Laboratory Forms binder (a separate binder is maintained for lead forms and asbestos/mold forms), in order that they might be copied and used on an as-needed basis (see section the Controlled Document Policy – section 5.2.4 of this Manual).

5.2.3.4 Correlation of Technical & Quality Assurance Records to Specific Chains of Custody

In most instances, all technical and quality assurance records associated with a particular job are maintained with that job throughout its progress in the laboratory and are stored with that file. However, in the case of metals analysis, samples are not necessarily analyzed on a per COC basis, but rather as an SDG, which may contain multiple COCs. In order to obtain a complete analytical record for any metals COC, the following procedures shall be followed.

1) From the AMA Search, look up the specific COC number in question. Print off and/or save the internal file in question.

2) Under the metals section of the AMA database, look up the COC number in question to obtain the SDG number.
3) Go to the SDG folder on the server and search for SDG number in question. The SDG file will contain the SDG summary, AA Log Data, and AA Raw Data. Print off and/or save the file question.
4) The combination of these two files (COC Internal & SDG) should produce a complete analytical record.

5.2.3.5 Backing up of Files

All critical files and databases are stored on the server. The server is backed up every business night to a tape. The tape is changed every business day and taken off site.

5.2.4 Controlled Documents Policy

This section addresses the control of raw data sheets and internal quality forms/password protected electronic documents/password protected software/software/analytical and preparation methods/laboratory accrediting agency manuals and other laboratory reference materials/instruction and operation manuals for laboratory instruments

The laboratory maintains a Master List of Controlled Documents in spreadsheet format (Appendix B of this Manual), which details the titles, revision numbers, and issue dates for all of the above referenced material. The documents that appear on this list are the most current version in use. Documents are reviewed during internal audits (see section 2.11 of this Manual) and, where necessary, revised to ensure continuing suitability and compliance with applicable requirements. Once a document has been revised, it is reviewed by the technical manager of the division that will use the document and/or the laboratory director. The approving manager indicates his/her approval by signing or initialing in the footer of the document. All computerized documents are either password or cell protected. Only personnel authorized to make changes to any computerized documents are in possession editorial passwords. When changes are made, those changes are tracked by using the word processing program's track changes command. After changes are made, documents are reviewed by the technical manager of the division that will use the documents and/or the laboratory director. The Master List of Controlled Documents is then updated to include the new revisions and/or document. Finally, the revised/new document is issued to all necessary personnel in a manner that details the changes made.

The previous version of any revised document is moved to the obsolete section of the Master List of Controlled Documents, stamped as obsolete, and immediately removed from circulation and placed in the Obsolete Documents Directory so that they may be retained for either legal or knowledge preservation purposes are suitably marked. Any document not currently in use by the laboratory in one form or another is considered obsolete. All obsolete documents are marked as such and are moved to the obsolete section of the Master List of Controlled Documents. They are then marked as being obsolete, removed from circulation, and placed in the Obsolete Documents Directory.

5.2.4.1 Archived Information

The Obsolete Documents Directory is considered archived information. The Access logs for all archived files are contained within the metadata for the individual files.

5.3 Sample Storage, Disposition & Pollution Prevention

The following sections outline AMA Analytical Services policies, by test method, regarding the storage, disposition, waste disposal, and pollution prevention for samples submitted to the laboratory for analysis. Specific procedures for disposal of samples and materials are outlined and discussed in the "Laboratory Safety Manual" and in specific SOPs, as referenced below.

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in our laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice, and our company subscribes to this. For example, typical stock solution preparation procedures follow for 500-mL quantities, but for the purpose of pollution prevention, we prepare smaller quantities whenever possible. Concentrations are calculated based upon the weight of the pure element or upon the weight of the compound multiplied by the fraction of the analyte in the compound. Whenever feasible, our laboratory personnel use pollution prevention techniques to address their waste generation. We require that laboratory waste management practices be conducted in a manner consistent with all applicable rules and regulations. AMA Analytical Services, Inc. strives to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. When wastes cannot be feasibly reduced at the source, we attempt to use recycling as the next best option whenever possible.

5.3.1 Air Samples for Transmission Electron Microscopy

Transmission electron microscopy of air samples results in three components that must be properly stored or discarded:

- cassette and unused filter
- unused carbon-coated filter
- prepared sample grid(s)

5.3.1.1 Disposal of Cassette and Unused Filter

The cassette and unused filter will be stored for a minimum of 30 days after the final report is issued to client. The original cassette and unused filter remnant will be returned to the client, if requested. The cassettes and unused filter are not considered to be asbestos containing under the NESHAP definition, but AMA personnel will act prudently with the material. The laboratory safety program addresses the procedure for disposing of cassettes.

5.3.1.2 Storage and Disposition of Unused Carbon Coated Filters

The slides containing unused carbon coated filter materials will be saved for a minimum of three (3) years after receipt of the sample. The slides are classified as non-asbestos-containing material, and are discarded accordingly.

5.3.1.3 Prepared Sample Grids

The prepared sample grids, regardless if they were analyzed or not, will be archived and stored for a minimum of three (3) years. The sample grids are classified as non-asbestos-containing material and are discarded accordingly.

5.3.2 Air Samples for Phase Contrast Microscopy

5.3.2.1 Disposal of Cassettes and Unused Filter

The original air cassette and filter remnant will be saved for a minimum of 30 days after completion of PCM analyses. Original cassettes and filter remnants will be returned to the client upon request. After 60 days, cassettes will be discarded as non-asbestos-containing material. Disposition of these cassettes is in accordance with Section 5.3.1.1.

5.3.2.2 Mounted Slides

The mounted slides for PCM analyses will be saved until completion of the written report. After the final results have been reported to the client and quality control has been performed in accordance with the QA/QC schedule, the mounted slides can be discarded. The mounted slides are considered non-asbestos-containing material and can be discarded with the cassettes.

5.3.3 Storage and Disposition of Bulk Asbestos Materials

All bulk materials submitted to the laboratory for asbestos analysis are considered to be asbestos-containing material. As a result, bulk materials are to be handled, stored and disposed of in a safe manner in accordance with all federal, state and local regulations in order to prevent pollution.

5.3.3.1 Storage of Representative Bulk Samples

It is the policy of the laboratory to store the entire sample remnant, or a representative piece of the original sample (if the original sample was large in size) for a period of at least 90 days.

5.3.3.2 Disposal of Excess Bulk Material

Excess bulk material is disposed of by placing the material in a sealed zip-lock bag, which is subsequently discarded in trash bags clearly marked as *asbestos-containing material*. These specially marked asbestos-containing disposal bags are double bagged and sealed. A licensed asbestos disposal firm is under contract and disposes of the material in accordance with federal, state, and/or local regulations.

5.3.3.3 Storage and Disposition of PLM Slides and Waste Products

PLM slides are discarded as asbestos-containing material as outlined in the above section. These slides can only be discarded after quality control analyses have been performed in accordance with the QA/QC schedules. All waste products generated during the sample preparation (i.e., tissues) are also discarded as asbestos-containing material.

5.3.4 Lead Samples

All samples submitted to the laboratory are considered to be lead-containing materials unless analysis shows otherwise. Bulk materials are to be handled, stored and disposed of in a safe manner in accordance with all federal, state and local regulations in order to prevent pollution.

5.3.4.1 Storage of Representative Bulk Samples

It is the policy of the laboratory to store the entire sample remnant, or a representative aliquot of the original sample (if the original sample was large in size) for a period of at least thirty days.

5.3.4.2 Disposal of Bulk Materials

All bulk materials determined by analysis to be hazardous lead waste are disposed of in accordance with all federal, state and local regulations.

5.3.4.3 Sample Digestates

Sample digestates are retained for at least 30 days after results are reported to the client. A minimum of 250 ml of undigested TCLP extract shall also be retained for at least 30 days. Samples are archived by SDG Number. Digestates with low lead levels (below 5 ppm) may be raised to a pH level above 4.0 prior to in-house disposal. Digestates containing lead concentrations above 5 ppm, which are ready for disposal, are stored in 55-gallon waste drums. Wastes in these drums are considered hazardous and disposed of in accordance with all local, state, and federal guidelines.

5.3.5 Hazardous Materials and Chemicals

Certain chemicals and by-products used for asbestos analysis are considered hazardous materials. Such materials are to be handled and discarded in accordance with federal, state, and local regulations in order to prevent pollution. The Laboratory Safety Officer is responsible for ensuring that all chemicals and hazardous materials are handled and disposed of in accordance with regulations and/or the laboratory safety program.

5.3.6 Mold Bulk Samples

All bulk materials submitted are considered as mold-containing materials until analysis shows otherwise. Bulk materials are handled, stored, and disposed of in a safe manner and in accordance with any/all applicable federal, state, and local regulations.

5.3.6.1 Storage of Representative Bulk Samples

It is the policy of the laboratory to store the entire sample remnant or a representative piece of the original sample (if the original samples was large enough in size) for a period of three months.

5.3.6.2 Disposal of Excess Bulk Material

Excess material is placed in a sealed bag and disposed of in regular waste.

5.3.6.3 Storage and Disposal of Mold Slides and Waste Products

Mold bulk slides are placed in a sealed bag and discarded as regular waste. These slides may only be discarded after quality control analyses have been completed in accordance with the QA/QC schedules.

5.3.7 Storage and Disposition of Air Samples for Mold Analysis

5.3.7.1 Disposal of Cassette

The sample cassette is placed in a sealed bag and thrown away in regular waste.

5.3.7.2 Storage of Sample Slides

The sample slides are archived and stored for three months. After three months the slides are placed in a sealed bag and thrown away in regular waste.

5.3.8 Storage and Disposal of Swabs for Mold Analysis

Based on the sample preparation, swabs can be stored and disposed of two different ways.

- If a direct prep of the swab was made
- If an indirect prep (sonication) of the swab was made

5.3.8.1 Direct Prep of Swab

The swab is placed back in original container and then stored for three months. After a minimum of three months the swab is placed in regular waste.

5.3.8.2 Indirect Prep of Swab

This sample preparation process results in three components that must be stored and then disposed of properly.

- Sonicated Swab
- Unused Filter
- Prepared Sample Slide

5.3.8.2.1 Sonicated Swab

Once the swab has been sonicated it is placed in a sealed bag and thrown away in regular waste.

5.3.8.2.2 Unused Filter Material

The unused filter material is placed in a filter storage unit and stored for a minimum of three months. After at least three months the samples are thrown away in regular waste.

5.3.8.2.3 Prepared Sample Slide

The prepared slides will be archived in a slide box for three months. After three months the slides are placed in a sealed bag and disposed in regular waste.

5.3.9 Water Sample for Mold Spore Identification

The analysis of water samples results in three components that must be properly stored and then discarded.

- Unused Water Sample
- Unused Filter Material
- Sample Slide

5.3.9.1 Unused Water Sample

Once sample has been analyzed the unused portion of the water can be disposed of by pouring it down a sink.

5.3.9.2 Unused Filter Material

The unused filter portion of the filter will be placed in a filter storage unit and stored for three months. After three months the samples are disposed in regular waste.

5.3.9.3 Sample Slide

Sample slides will be stored for three months. After three months the sample slides will be placed in sealed bags and disposed in regular waste.

5.3.10 Plates for Viable Samples

Culture plates are stored for 3 months from the date of analysis. Prior to disposal, all culture plates are decontaminated in the autoclave in order to prevent pollution. For detail procedures and operation of the autoclave see SOP #911. Also see the Biosafety Plan for the Mold Laboratory.

5.3.11 Reference Materials

All reference materials and samples are stored and handled using the same safety and storage procedures used for routine samples. These materials differ from routine samples in that they are kept indefinitely for training and quality control purposes.

9.0 TEM ANALYSIS OF AIR SAMPLES FOR ASBESTOS

The purpose of this section is to define specific procedures for the analysis of air samples for asbestos in accordance with the AHERA methodology and the NIOSH 7402 method. Laboratory Technicians and Operators, as well as the Laboratory Supervisor, will follow the steps outlined in this section. Standard operating procedures (SOPs) pertaining to the AHERA and/or NIOSH 7402 asbestos analysis are referenced in this section. The specific SOPs are located in Appendix A of this manual.

9.1 Sample Preparation

The laboratory prepares both mixed cellulose ester (MCE) and polycarbonate (PC) filters for the analysis of airborne asbestos.

Direct preparation techniques are utilized for all samples.

9.1.1 MCE Preparation

The preparation of MCE filters follows the procedure shown in SOP No. 201.

9.1.2 PC Preparation

The preparation of PC filters follows the procedure shown in SOP No. 202.

9.1.3 Laboratory Blanks

A laboratory blank is prepared with every set of samples. Each lab blank is assigned a unique number. The laboratory blank is analyzed if the set of samples associated with the blank fails or if the lab blank number ends with a 0. This insures that at least one blank is analyzed for every 25 filter preparations. SOP No. 206 outlines the steps to be taken if asbestos contamination is found on the laboratory blank.

9.2 Preliminary Assessments

In accordance with the AHERA document 40 CFR Part 763, a response action is determined to be completed by TEM when the abatement area has been cleaned and the airborne asbestos concentration inside the abatement area is not higher than concentration at locations outside the abatement area. "Outside" means outside the abatement area, but not necessarily outside the building. A minimum of five air samples from inside the abatement area and a minimum of five air samples from outside the abatement area are collected; the response action is determined to be complete when the average airborne asbestos concentration measured inside the abatement area is not statistically different from the average airborne asbestos concentration measured outside the abatement area.

When volumes greater than or equal to 1,199 L for a 25 mm diameter filter and 2,799 L for a 37 mm diameter filter have been collected and the average number of asbestos structures on samples inside the abatement area is not greater than 70 s/mm^2 of filter, the response action is considered complete. This screening test is not used when volumes of less than 1,199 L for 25 mm diameter filter or 2,799 L for a 37 mm diameter filter are collected because the ability to distinguish levels significantly different from filter background is compromised (reduced) at low volumes.

The initial screening test is expressed in structures per square millimeter (S/mm^2) of filter. The value of 70 s/mm^2 is based on the background asbestos contamination on "blank" filters. Calculations to determine filter area to be analyzed are shown in section 9.2.3.

The inside and outside concentrations are compared by the Z-test. The Z-test is a statistical calculation that takes into account Poisson distributed events and the variability in the measurement process of rarely occurring events. A minimum of five samples inside the abatement area and a minimum of five samples outside the abatement area are required to control the false negative error rate, i.e.: the probability of an Industrial Hygienist (IH) consultant declaring the removal complete when, in fact, the air concentration inside the abatement area is significantly higher than outside the abatement area. Additional quality control is provided by requiring three blanks (filters through which no air has been drawn) to be analyzed to check for unusually high filter contamination that would distort the test results.

Interpretation of Results:
The response action shall be considered complete if either:

a. Each sample collected inside the abatement area consists of at least 1,199 L of air for a 25 mm diameter filter, or 2,799 L of air for a 37 mm diameter filter, and the arithmetic mean of their asbestos structure concentrations per square millimeter of filter is less than or equal to 70 s/mm^2, **OR**

b. The arithmetic mean of the asbestos structure concentration on the three blank filters is less than or equal to 70 s/mm^2 and the average airborne asbestos concentration measured inside the abatement area is not statistically higher than the average airborne asbestos concentration measured outside the abatement area as determined by the Z-test.

The Z-test is carried out by calculating $Z=[Y(I)-Y(O)] / [0.8*(1/n(I) + 1/n(O))^{1/2}]$ where Y(I) is the average of the natural logarithms of the asbestos concentration (in s/cc) of the inside samples and Y(O) is the average of the natural logarithms of the asbestos concentration (in s/cc) of the outside samples, n(I) is the number of inside samples and n(O) is the number of outside samples. The response action is considered complete if Z is less than or equal to 1.65.

(Note: For Z-test calculations, when no fibers are counted, the calculated analytical sensitivity is inserted for the concentration.)

9.2.1 Quality Check

Client sample designations and corresponding air volumes are compared to identifications supplied by the client. If discrepancies exist, a senior analyst is notified. Clients are contacted for clarification as necessary.

9.2.2 The Count Sheet

A new Count Sheet is used for each sample to be analyzed. A copy of the Count Sheet is located in the Forms section of this manual.

The Count Sheet is logically designed in three sections.

Section 1: Top - Details:
- Laboratory name
- Date of analysis
- Name and signature of the Operator
- Client's sample number
- Corresponding AMA sample number

Chain of custody number
Grid box label
Grid lot number
Grid location coordinates
Microscope information
Sample volume
Filter type, manufacturer, and lot number
# of grid openings to analyze according to AHERA
Sensitivity of analysis
Orientation of the letter F.
Grid acceptable

Section 2: Central - Details:
Grid coordinates
The identity of the 2nd grid
Structure number
Structure type
Structure length and width
SAED results
Spectrum ID label, if acquired
Identified elements
Negative number of a fiber image (brightfield)
Negative number of an SAED pattern
Measurement units of the photographs

Section 3: Bottom - Details:
Total # of grid openings observed
Mean grid area
Total observed area
Total # of countable structures observed by size
Total # of asbestos structures observed and concentration by size
# of asbestos structures observed by type, size and concentration
# of non-asbestos structures and concentration
Calibration dates for camera constant, magnification and EDXA
Spectrum floppy disk label
TEM serial number
Laboratory address, phone number and facsimile number

9.2.3 Analysis Area Determination

Guidelines for the Determination of the Area to Analyze

Grid Openings to Analyze :

$$\frac{\text{Effective filter area}}{\text{Sensitivity X grid opening area X Volume sampled X 1000}}$$

Then round up to the next integer.

Analyzed half of the area to be analyzed on the first grid and the other half on the second grid.

Guidelines for Determination of Analytical Sensitivity:

Analytical Sensitivity: (s/mm^2) $\quad \frac{1}{\text{Area Analyzed}}$

Then round up to the next integer

Analytical Sensitivity: (s/cc) $\quad \frac{\text{Effective filter area}}{\text{Area Analyzed X Volume X 1000}}$

Then round to four decimal places.

9.2.4 TEM Description and Settings for Analysis

The JEOL 100CXII microscopes operate at 100kV and is capable of performing electron diffraction with a fluorescent screen inscribed with calibrated circles of 74mm and 7.4mm diameter, and a linear scale from 0.0 to 16mm. The TEM is equipped with an EDXA spectrometer and is capable of producing an electron beam spot size less than 250nm in diameter at crossover, and an electron diffraction pattern of single fibers of chrysotile. Each microscope is equipped with both single and double-tilt holders for electron diffraction pattern analysis. It is capable of recording brightfield and electron diffraction patterns on electron image film. It has a mechanical stage with linear, reproducible movements along two perpendicular directions. The microscope is calibrated monthly for magnification (at 19k, 14k, and 2.9k), spot size (using spot size 4) and camera constant.

Microscope Settings Used for Analysis:

* 100 kV
* grid assessment 100x-300x working magnification
* ca.15,000x screen magnification for analysis
* ca.19,000x film plane magnification

* C1 setting at spot size 1 (brightfield analysis)
* C1 setting at spot size 2 through 4 for EDXA

9.2.5 TEM Alignment

TEM alignment is checked by the operator prior to analysis. (see SOP 301 entitled, Alignment Procedure) (For beam dose measurement, align the microscope according to SOP 301,and use the microscope settings for routine analysis)

9.2.6 Grid Orientation and Traverse

Approximately one-half of the predetermined sample area to be analyzed is performed on one sample grid preparation and the remaining half on a second sample grid preparation.

Placement of the grid in the TEM, grid opening selection and traverse direction are outlined in SOP No. 302, Grid Opening Orientation, Selection and Traverse.

9.2.7 Prepared Grid Evaluation

The prepared grid will be considered acceptable for analysis provided the following conditions are met.

At low magnification (100-300x) viewing:

1. Greater than 50% of all grid openings observed are covered by the carbon replica, whether or not the covered openings are electron transparent.
2. Greater than 50% of the grid openings covered by the replica are intact.
3. Less than 10% of the filter area covered by the replica is undissolved.
4. Less than 50% of the grid openings covered by the replica have evidence of overlapping or folded replica film, with at least 20 grid openings having no overlapping or folded replica.
5. The particulate loading on the replica is less than 10%.
6. The overall particulate loading appears to be evenly distributed over the entire replica.

For individual grid openings covered by the replica the following conditions are met:

1. Less than 5% of the filter area is undissolved/opaque.
2. Holes comprise less than 5% of the replica.

9.3 Structure Identification

During the analysis of air filter samples by TEM, structure identification guidelines are necessary. TEM operators use these guidelines to make decisions regarding the identification of an asbestos structure.

Specific parameters for the identification of asbestos structures are outlined in this section. These parameters are:

- Definition of Structures (9.3.1),
- Structure Measurement (9.3.2),
- SAED Analysis (9.3.3), and,
- EDXA (9.3.4)

Mineral types that may be encountered by an operator which are not asbestos and may be mistaken for asbestos are discussed in section 9.3.5, Non-Asbestos Minerals.

Guidelines for recording data and stopping the analysis are also outlined. These guidelines are discussed in sections 9.3.6, Stopping Rules, and 9.3.7, Recording Rules.

9.3.1 Definition of Structures

Structures are defined according to the categories outlined in AHERA (40 CFR Part 763, Appendix A - Subpart F).

Essentially, any individual fiber having an aspect ratio greater than or equal to 5:1 and a length greater than or equal to 0.5 micrometers (um) and substantially parallel sides, or any continuous grouping of particles in which a fiber is detected, shall be characterized as a structure. Moreover, the identified structure is classified as follows:

Asbestos Structure Classification	***Criteria***
Fiber	A structure having a minimum length greater than or equal to 0.5 um and an aspect ratio (length to width) of 5:1 or greater and substantially parallel sides. The appearance of the end of the fiber, (i.e., whether it is flat, rounded or dovetailed) is noted.
Bundle	A structure composed of three or more fibers in a parallel arrangement with each fiber, closer than one fiber's diameter.
Cluster	A structure with fibers in a non-parallel arrangement such that all fibers are intermixed and no single fiber is isolated from the group. Groupings must have more than two intersections.
Matrix	Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the fiber definition.

(The guidelines for measuring a fiber are shown in section 9.3.2, Structure Measurement.)

If the fiber meets the size and aspect ratio definition as a structure, then the structure is classified as above. The structure type is entered onto the Count Sheet following the guideline shown in section 9.3.7, Recording Rules.

9.3.2 Structure Measurement

Structure measurement is completed on the TEM using the calibrated scales on the fluorescent viewing screen. (See SOP No.404, Magnification Calibration.)

The operator will orient (using the stage controls) the longest fiber within a structure along the inscribed scales and visually estimate the length and width of the fiber. Measurements (in micrometers) are entered onto the count sheet according to the Recording Rules shown in section 9.3.7.

Structure identification using SAED and/or EDXA is then completed. (Refer to sections 9.3.3 and 9.3.4, respectively).

When the measurements and identification are completed, the structure is repositioned to its original location on the viewing screen using the stage control.

The sample analysis for that grid opening then continues in the same traverse direction.

9.3.3 Selected Area Electron Diffraction (SAED) Analysis

Overview

Selected area electron diffraction (SAED) analysis is used to characterize a structure's crystallography. Specifically, the coherent scattering of electrons interacting with a crystal (e.g., "asbestos") produces a pattern on the fluorescent viewing screen or film. The pattern produced is unique for the crystal system analyzed.

Crystal systems for common varieties of asbestos encountered are as follows:

Crystal System	Asbestos
Monoclinic	Chrysotile, Amosite, Tremolite, Actinolite, Crocidolite
Orthorhombic	Anthophyllite

SAED Pattern Generation

SAED patterns are obtained and photographed following the procedure outlined in SOP No. 303, How to Get a Diffraction Pattern on the TEM.

Film development to produce negatives is outlined in SOP No. 305, Photo Processing and Dark Room Procedures.

SAED Indexing

The detailed procedure for SAED pattern indexing is shown in SOP No. 304, SAED Indexing Procedure.

A description of the SAED patterns for chrysotile and amphibole minerals is shown below.

Chrysotile

The chrysotile SAED pattern is unique due to the "scrolling" effects of the phyllosilicate construction.

The pattern has characteristic streaks on the layer lines. (The central layer line may not exhibit complete streaking.) There are diffraction spots of normal sharpness on the central layer line and on alternate lines (i.e., 2nd, 4th, etc.). The repeat distance between layer lines is 5.3 Angstroms and the center doublet (002) is 7.3 Angstroms.

The form used for indexing suspected chrysotile patterns is shown in the Forms section.

Amphiboles

Unlike chrysotile, the amphiboles will produce different SAED patterns depending on the zone axis detected. In general, the amphibole group (including the asbestiforms, actinolite, amosite, anthophyllite, crocidolite and tremolite) produces SAED patterns with layer lines formed by closely spaced diffraction spots and a repeat distance between layer lines (and on alternate lines -- 2, 4, etc.) of 5.3 Angstroms.

Layer line streaking is usually not encountered for the amphiboles, but due to structural defects may occur.

The forms used for indexing the monoclinic amphiboles and the orthorhombic (anthophyllite) amphibole is shown in the Forms section.

Evaluation Criteria

Acceptance criteria for SAED patterns consistent with asbestos are based on the measured d-spacing values as compared to the values shown in the JCPDS powder diffraction files and Dr. Shu-Chun Su's reference tables for asbestos mineral zone axis patterns . Unknown patterns that are within plus or minus 5% of the d-spacing values shown in the JCPDS for asbestos minerals are considered consistent with asbestos. Values measured outside the 5% limitation are considered not consistent with asbestos.

For complete identification of the asbestos encountered, the energy dispersive X-ray analysis (EDXA) spectrum for the structure is obtained. (See section 9.3.4.)

SAED Pattern Indexing Frequency

Visual identification of SAED patterns and/or indexing is completed for each suspected asbestos structure encountered which causes an analysis to exceed the 70 s/mm^2 concentration for each AHERA "inside" sample set. This is normally the first five asbestos structures per sample.

If upon completion of the analysis for an AHERA "inside" sample set, the average structures detected are less than 70 s/mm^2, the SAED indexing may be completed after reporting results (i.e., <70 s/mm^2) to the client.

SAED analysis may be used without EDXA spectroscopy to confirm the presence of chrysotile after 70 s/mm^2 concentration has been exceeded.

9.3.4 Energy Dispersive X-Ray Analysis (EDXA)

Overview

EDXA is used to characterize a structure's chemistry (i.e., the elemental component). Specifically, X-rays are produced through the interaction of the incident electron beam with a particle (crystalline or non-crystalline). A spectrum is produced which indicates the elements detected for that particle.

Limitations of EDXA spectroscopy are based on the concentration and atomic number of an element within the particle, orientation relative to the detector, and the resolution of the detector.

AMA Analytical Services' EDXA systems detect elements greater than an equal to Z = 11 (sodium) on scope 1 and Z = 6 (carbon) when present in quantities greater than 1% and within the line-of -sight of the detector.

Asbestos Chemistry

The following table shows the range of percentages of elements for each asbestos type.

Compound	Tremolite	Crocidolite	Amosite	Anthophyllite	Actinolite
Si0	49-53	49-53	56-58	51-56	55-60
MgO	0-3	1-7	3-12	15-20	21-26
FeO	13-20	34-44	28-34	5-15	0-4
$Fe_2 0_3$	17-20	-	-	0-3	0-0.5
$Al_2 0_3$	0-0.2	-	0.5-1.5	1.5-3	0-
CaO	0.3-2.7	-	-	10-12	11-13
$K_2 0$	0-0.4	0-0.4	-	0-0.5	0-0.6
$Na_2 0$	4.0-8.5	-	-	0.5-1.5	0-1.5
$H_2 0$	2.5-4.5	2.5-4.5	1.0-6.0	1.5-2.5	0.5-2.5

Compound	Chrysotile
$Si0_2$	38.8-41.5
$Fe_2 0_3$	0.04-1.6
Fe0	0.3-2.0
$Al_2 0_3$	0.04-4.7
Mg0	38.2-42.6
Ca0	0.35-2.0
$Na_2 0$	0.04-0.1
$K_2 0$	0.02-0.2
$H_2 0$	12-13.8

Standard reference spectra for each asbestos type are maintained in the laboratory.

Detector Quality

The EDXA spectrometer is calibrated daily for Al and Cu peak energy values in accordance with SOP Nos. 407 and 408, Kevex and Thermo-Noran Calibration.

The K-alpha peak resolution (at FWHM) for manganese is 175 eV or lower with a value of sum of the resolution and the variation (2 s) is less than 180 eV, and calibrated at least quarterly (or after maintenance service).

Asbestos Identification Criteria

Asbestos is identified through a comparison of an EDXA spectrum for an unknown structure to known asbestos standards. If the proper elements are detected and the peak heights (indicative of quantity) approximate known standards, then the structure may be identified.

EDXA Spectroscopy, Frequency of Analysis

EDXA spectroscopy is completed for most suspected amphibole asbestos structure encountered.

EDXA spectroscopy is completed on suspected chrysotile structures when SAED is not obtainable.

EDXA may be used without SAED analysis to confirm the presence of asbestos after 70 s/mm^2 concentration has been exceeded. EDXA is also used without SAED analysis to confirm non-asbestos identification.

9.3.5 Non-Asbestos Minerals

During the analysis of air samples many non-asbestos minerals may be encountered. The TEM operator uses morphology, SAED and EDXA criteria to identify the mineral as asbestos or not asbestos.

In some cases (e.g., gypsum - calcium and sulfur rich), EDXA alone is sufficient to identify a structure as a non-asbestos mineral.

The following list of minerals are potentially confused with asbestos. The guidelines following each mineral aid the TEM operator in identifying the unknown structure and to differentiate it from the regulated asbestiforms. (The listing is a summary of a presentation by James Smith, Georgia Institute of Technology, 1989, "Interpretation of the NVLAP Requirements for TEM in Regard to Interfering Minerals...")

Mineral Type	Guideline
Pyroxenes	The repeat pattern for the d-spacing along the b-axis is one-half that of amphiboles (approximately 9 Angstroms).
Halloysite	An aluminum silicate. EDXA will show its difference from chrysotile. Although tubular and yielding a similar SAED pattern to that of chrysotile, the tube is coincident with the b axis; in the most common form of chrysotile, the 2MVl form, the tube is coincident with the a axis (parallel to the fiber's length). (From Gard, 1971)

Palygorskite	Aluminum is common in palygorskite. Aluminum is not present in the regulated asbestiforms. Palygorskite is non-cylindrical, so its SAED pattern will not appear streaked (unless interference occurs). The [100] d-spacing is 12 Angstroms.
Sepiolite	EDXA can distinguish sepiolite from chrysotile by its Mg:SI ratio: 1.3 in chrysotile, 0.4 in sepiolite. Sepiolite does not have a cylindrical lattice; its SAED pattern will not be streaked. The (010) reflection in sepiolite is 26.9 Angstroms, versus the (020) spacing of the amphibole's 9.5 Angstroms.
Antigorite	A polymorph of chrysotile, the chemistry is similar. Its SAED yields a modulated structure that creates a superlattice, varying from 25 to 50 Angstroms, causing a grouping of reflections
Lizardite	Another polymorph of chrysotile, its chemistry is also identical. Its SAED pattern will exhibit a hexagonal array of reflections (common to the phyllosilicates), impossible to view in the regulated asbestiforms unless the beam travels down the c axis. (The fiber would have to be very short and perpendicular to the replica.)
Talc	Mg:Si ratios differentiate from chrysotile. Talc is typically 0.6. Talc is also a phyllosilicate that does not scroll (normally). Therefore, it exhibits a hexagonal SAED pattern, which does not have streaked reflections.
Vermiculite	An aluminum-rich phyllosilicate, it is differentiated from the regulated asbestiforms by its EDXA.

A notebook listing crystallographic, elemental, elemental standard deviations, and morphological details of all the asbestos types and common non-asbestos look a likes is located in scope room #1. The chemical data and photographs were collected on scope #1.

9.3.6 Stopping Rules

The TEM operator follows these guidelines when terminating an analysis of an AHERA sample set. The analysis is terminated when:

- The area observed for the analysis is sufficient to have an analytical sensitivity of less than or equal to 0.005 s/cc and a one fiber value of 17 s/mm^2.
- The 50th asbestiform structure is counted in a single grid opening.
- The grid opening containing the 50th asbestos structure is completed.

- During the analysis of a blank sample, a one-fiber value of 17 s/mm is an achieved.

9.3.7 Recording Rules

A description of the Count Sheet used during the analysis of TEM air samples is shown in section 9.2.2. A copy of the "Count Sheet for Transmission Electron Microscopy" is shown in the Forms section of the Appendices.

All information regarding the sample identification is shown in Section 1 of the Count Sheet (e.g., laboratory name, date of analysis, AMA sample number, client sample number, etc.). All information in Section 1 of the Count Sheet is recorded by the TEM operator. Discrepancies are discussed with the TEM Laboratory Supervisor and/or the client.

The central (Section 2) portion of the Count Sheet provides detailed data regarding the analysis of a TEM air sample. Specifically, data pertaining to the analysis is entered beneath the named columns as follows:

Grid Opening
/ Location

The indexed grid opening location is provided in the "main" portion of this column. For example, if the analysis begins with the grid opening P3, then P3 is entered into the box.

A smaller box in the upper right hand corner is to be left open during the analysis of any grid opening analyzed from the first grid preparation for a sample; an **X** is placed into the box for analysis of grid openings from a subsequent (second or third) grid preparation for a sample.

Alternatively, the operator may enter an a, b, or c to denote the 1st, 2nd or 3rd grid preparation.

Structure
Number

A sequential numbering (beginning with "1") of all structures detected meeting the definition of structures (see Section 9.3.1) is entered into this box. The Structure Number increases in increments of 1 for the analysis of an entire sample. (The numbering does not begin at "1" for each grid opening.)

If no structures are detected for a grid opening, then "NSD" is entered.

Structure Type

Structures are defined according to their type (e.g., fiber, bundle, etc., see Section 9.3.1) and entered in accordance with the legend at the bottom of the Count Sheet.

Length and Width

Structures are measured according to Section 9.3.2, Structure Measurement. Values (in microns) for the length and width of a structure are entered.

SAED

After a visual SAED analysis of a structure, "POS" is entered for a positive visual identification for chrysotile asbestos and the number of rows visible in the calibrate aperture for amphibole asbestos; "NEG" is entered for a negative visual identification for asbestos.

If a pattern is not obtainable, then "UTO" is entered (as per the legend). If SAED analysis is not attempted the space is left blank.

SAED pattern indexing is completed in accordance with Section 9.3.3, SAED Analysis, at a rate specified in the heading entitled SAED Pattern Indexing Frequency.

Saved Spectrum

Spectra are saved to a file based on the chain of custody number of the set. Spectra are only saved on scope #2.

If an EDXA spectrum is not obtainable, then "UTO" is entered. If an EDXA analysis is not attempted the space is left blank.

Elements Identified

Each element identified for a structure by EDXA analysis is entered.

Fiber Negative Number

The unique identification number ascribed to a TEM image of a structure and appearing on the negative is entered.

SAED Negative Number

The unique identification number ascribed to an SAED pattern appearing on the negative is entered.

Magnification or Camera Length

For all photomicrographs taken of structures, the magnification is entered.

For all SAED patterns photographed the camera length is entered.

The bottom (Section 3) portion of the Count Sheet summarizes data recorded in the central portion. Also, information regarding the calibration of the microscope and EDXA system is entered as well as grid opening area data, spectra disk ID# and the serial number of the microscope.

If the analysis of a sample requires the use of multiple Count Sheets, then the page number record is entered in the upper right hand corner. Furthermore, page 1 of the analysis will show complete information for the top and bottom portions of the count sheets. The operator is only required to enter the client name, sample number and AMA Lab ID Number in the top portion of an analysis for pages 2 through n for a sample (as well as all data records shown in the central portion).

9.4 Reporting

Upon completion of the analysis, the Operator calculates the size distributions of all counted structures, and calculates the asbestos and non-asbestos concentrations and records them at the bottom of the first page of each sample's count sheet(s).

REPORTED CONCENTRATION: $\frac{\text{\# Asbestos Structures Detected}}{\text{Area Analyzed}}$ s/mm^2

Then, round up to the next integer.

If less than 1 asbestos structure is detected, use 1 in the numerator and report as " <____ " s/mm^2. Otherwise report as the actual value.

REPORTED CONCENTRATION: $\frac{\text{385 X \# Asbestos Structures Detected}}{\text{Area Analyzed X Volume X 1000}}$ s/cc

Then round mathematically.

If less than 1 asbestos structure is detected, use 1 in the numerator and report as " <____ " s/cc. Otherwise, report as the actual value.

When the analysis of a group of samples has been completed, the analyst enters the results into the LIMs system and prints out a certificate of analysis, invoice, and cover page.

Results are reported verbally, by facsimile, by e-mail, or by voice mail. Whenever possible, any associated QC analysis and an independent data review by a qualified person should be completed and the technical manager or his designee should place their finalizing signature on the Certificate of Analysis prior to reporting results to the client. All data is considered preliminary and subject to change until the technical manager or his designee has signed the Certificate of Analysis. The signature of the technical manager indicates that all associated QC and an independent data review by a qualified person has been completed to the satisfaction of the technical manager. After generation of the Certificate of Analysis, the signed document is mailed to the client.

9.4.1 Final Report

The certificate of analysis produced by the analyst is the final report that is mailed to the client.

The following information is reported to the client for each sample analyzed:

1. Concentration in structures per square millimeter and structures per cubic centimeter.
2. Analytical sensitivity used for the analysis.
3. Number and types of asbestos structures. Number and types of non-asbestos structures.
4. Area analyzed.

5. Volume of air sampled (which is supplied by the client).

6. Copy of the count sheets which contain the detailed description of each fiber observed, and the breakdown of concentrations and total number of structures by size.

7. Signature of analyst to indicate that specifications of the method are met.

8. The reports are printed on official laboratory letterhead.

Data generated is filed for an indefinite period of time. The current method of storing records is done by scanning all documents associated with a chain of custody into a PDF file. The originals are shredded after being scanned. This method is used for all records after October 1, 2002. The originals of all records prior to this date are stored for a period three to five years depending on whether the accrediting agency is ELAP or NVLAP.

Once generated and reported, data written on the certificate cannot be amended without consultation with the Laboratory Director and/or the Client. If possible the original certificate is retrieved or destroyed prior to the release of the amended certificate. Copies of certificates must be clearly labeled as such.

9.5 Analytical Quality Control of TEM Air Samples

The LIMS system is used to assign replicate and duplicate QC samples monthly as outlined in section 16.6.3 of this manual. All replicate and duplicate QC is completed prior to mailing the final report. The following procedures are used to maintain a high level of quality control on TEM air sample analysis.

1. Verified Count Analysis - Verified count analysis is performed by each TEM analyst on samples with known asbestos concentrations at a rate of 1 per every 100 grid openings analyzed. Each analyst must maintain an 85% true positive level to remain qualified to analyze TEM samples. If an analyst falls below an 85% true positive level additional training is given until that analyst re-qualifies. The NIST 1876b is issued at least once per year for verified count analysis. The data is summarized per analyst and stored in the Quattro Pro verified count spreadsheet.

2. Replicate Analysis - Five percent of all air samples analyzed by each individual analyst are reanalyzed by another analyst. The replicate analysis is completed prior to mailing the final report.

3. Duplicate Analysis – Two to Three percent of all air samples analyzed by each individual analyst are reanalyzed by the same analyst. The duplicate analysis is completed prior to mailing the final report.

4. Blank Analysis - A blank sample is prepared along side with each set of samples. If the results of any of the live samples within the sample set is above the clients release criteria the blank sample is analyzed to check for possible contamination. Blank samples that have a number ending in zero are also analyzed.

5. Re-preparation of filters – Three samples are randomly selected and prepared a second time for re-analysis per month. The preparations are compared to the original sample preparations. Particulate loading, plasma ashing texture, fiber distribution, and the overall quality of the preparations are qualitatively evaluated. The analyst records their observations in the LIMMs system, specifically in the comments section of the replicate or duplicate TEM air form.

9.5.1 Correction of Analytical Errors

All of the replicate and duplicate QC is completed prior to the mailing of the final report, however, verbal or faxed results are reported immediately after analysis. If QC sample analysis changes an original result in a way that effects the final concentration of a sample or a set, changing the sample or set from pass to fail, the client is immediately notified of the change. A new report is printed reflecting the change in results.

9.6 NIOSH 7402 Analysis

The following section outlines the procedures used when analyzing a NIOSH 7402 sample.

9.6.1 Sample Preparation

Follow the procedures outlined in SOP 201 but skip the plasma etching steps.

9.6.2 Sample Analysis

The following guidelines are used when analyzing NIOSH 7402 samples.

Overloading Criteria: The method allows for greater than 50% particulate loading across the entire grid before overloading the sample. The restriction on individual grid openings is 20%. So for example a sample with 40% particulate loading may or may not be overloaded. If the sample has large particulate that clumps some of the grid openings may be 50, 60+ percent covered with particulate while others are only 5-10%. In this situation pick 40 grid openings with less that 20% particulate over all 3 grids. If there are not 40 with less than 20% the sample is overloaded. If the particulate was small and evenly distributed there most likely won't be any grid openings with less than 20% particulate.

Analysis: Using a screen magnification of 1,500x Analyze 40 grid openings over 3 grids. Count fibers longer than 5 microns with a diameter of >0.25 microns and an aspect ratio of >=3:1. Count fibers sticking out of grid bars as half fibers provided that more than 2.5 microns of the fiber is visible.

Stopping rules: This method has a 100 fiber with a minimum of 6 grid openings stopping rule. You must complete the grid opening that contains the 100^{th} fiber. You cannot stop in the middle of a grid opening.

9.6.3 Reporting

Use the same procedures for reporting as those outlined in section 9.4 of this manual with the following exceptions:

Use a 4 fiber LOD (limit of detection) when calculating results. If less than 4 asbestos fibers are observed report the sample as less than 4 times the analytical sensitivity.

Report in f/cc as opposed to s/cc.

13.0 TEM QUALITY CONTROL PROCEDURES

The purpose of this section is to define the specific quality control procedures relative to analysis of NOB samples, TEM air samples, TEM water samples, and the equipment used in the preparation and analysis of these samples. Laboratory technicians and analysts will follow the steps outlined in this section. The QC supervisor conducts an audit and review of all laboratory procedures twice a year, in April and October, to insure that the laboratory is compliant with all NVLAP and ELAP requirements. A quality control meeting is held on the second Friday of each month to review and summarize the previous months QC.

13.1 Analysis Equipment Quality Control and Maintenance

One JEOL 100CX II electron microscope with a KEVEX X-Ray detector is used during analysis. The other JEOL 100CXII is equipped with a Thermo-Noran Quest 1 X-ray system. Numerous calibrations are performed on this equipment at various time intervals including:

1. Copper Aluminum Calibration of the Kevex Detector - Performed daily or at the beginning of each day that scope is in use. The Quest system only requires one peak to calibrate the system. Any peak in the spectrum may be used, however, copper is the peak typically selected.

2. Screen Magnification Calibration - Performed weekly on the first day of each business week.

3. Photo Plate Magnification Calibration - Performed weekly on the first day of each business week.

4. Camera Constant Calibration - Performed weekly on the first day of each business week.

5. Spot Size Diameter - Performed weekly on the first day of each business week.

6. Beam Dose Check - Performed monthly.

7. Detector Resolution Check - Performed monthly.

8. Sodium (Na) Detection Check - Performed monthly.

9. Magnesium (Mg) Detection Check - Performed monthly.

10. K-Factors - Performed quarterly and immediately after a new crystal is installed. The NIST 2063a is used for Mg, Fe, and Ca K-Factors. A characterized albite is used for the Al K-Factor.

11. Muffle Furnace - Both of the ovens are calibrated during the first week of each month. The results are recorded in a spreadsheet. The thermometer used to record the temperatures is check prior to use by recording the temperature of boiling water.

If as result of a QC procedure a problem is found with a piece of equipment the manufacturer or service technician is contacted and servicing of the equipment is performed. The data generated from each quality control procedure is kept on computer spreadsheets and control charts are generated to track results over a long-term basis.

13.1.1 Control Charts

Whenever applicable, control charts are used to track the performance of laboratory equipment. Initially, default control limits are used to assess equipment performance. After sufficient measurements of the operating parameter have been collected (at least 25 measurements), the control limits for the equipment are adjusted by computing a running mean and standard deviation of the operating parameter measurement (i.e. TEM photo plate magnification). The upper and lower acceptable limits are set at +/- 3 standard deviations from the running mean, or are kept at the default control limits, whichever are stricter. Warning limits of +/- 2 SD from the mean may also be used to detect changes in performance before control limits are exceeded. The setting of warning limits, and adjustments to the control limits are made by the Quality Control Supervisor for the purpose of detecting changes in the operation of equipment in a timely manner. Regular evaluation of the most recent measurements using control charts with tight control and warning limits enables the Quality Control Supervisor to spot changes in operating performance of equipment before it becomes a serious problem which may effect the quality of analytical results.

13.2 Preparation Equipment Calibration and Maintenance

13.2.1 Hoods

The vacuum hood and HEPA filter hood are wet-wiped daily with fiber-free water. Airflow is measured twice a year by the corporate QC officer and shall be 80 to 120 linear feet per minute. The HEPA filter is replaced whenever minimum acceptable airflow cannot be maintained or every two years, whichever comes first. The HEPA filters are disposed as asbestos containing waste.

13.2.2 Plasma Asher

Calibration of the plasma asher is performed photographically.

The plasma asher chamber is cleaned after each use with fiber-free water and dried with a lint-free cloth.

Pump oil is monitored weekly for signs of degradation. Oil is changed annually or as necessary. Vacuum tubes are changed as necessary.

13.2.3 Carbon Coater

Mechanical pump oil and diffusion pump oil are changed annually or as needed. The Bell jar is cleaned weekly or as needed.

The Bell jar gasket is changed biannually or as needed.

13.2.4 Glassware and Miscellaneous Utensils

All glassware and miscellaneous utensils (i.e., stainless steel mesh, forceps, scalpels, etc.) are cleaned using detergent, solvents (e.g., acetone or ethanol) and/or fiber-free water.

(See SOP No. 205, Cleaning Stainless Steel Screens.)

13.2.5 Muffle Furnace

Muffle Furnace Calibration - The muffle furnace temperature is check with a temperature probe quarterly at temperatures of 400 C, 450 C, 480 C, 500 C, and 600 C. The probes accuracy is checked in boiling water.

13.2.6 Analytical Balance

The calibration of the analytical balance is checked weekly with two weights, 0.100g and 0.300g, at three tare levels: untared (empty pan), tared with an NOB vial (approximately 8 grams), and tared with a small glass beaker (approximately 60 grams). In addition, a daily check with the 0.300 gram weight is performed each day the balance is used.

13.3 Analytical Quality Control of TEM Bulk Samples

All replicate and duplicate QC is performed prior to mailing the final report. The following procedures are used to maintain a high level of analytical quality control on TEM bulk sample analysis:

1. Replicate Analysis of the PLM Preparation - At least one sample or ten percent of the samples, which ever comes first, of each set of NOB or friable samples is reanalyzed by another analyst.

2. Replicate Analysis of the TEM Preparation - One of every 15 NOB, friable TEM, or residual ash samples is reanalyzed by another analyst. The replicate analysis is completed prior to the mailing of the final report.

3. Duplicate Analysis of the TEM Preparation - One of every 50 NOB, Friable TEM, or residual ash samples is reanalyzed by the original analyst. The replicate analysis is completed prior to the mailing of the final report.

4. Blank Sample Analysis - A sample of known negative floor tile is prepared along side each set of NOB or friable samples. At least one blank sample for every 20 samples is analyzed.

5. Reference Sample Analysis – A reference NOB sample is prepared with every batch of samples. The reference sample is randomly selected from our reference library. Upon completion of analysis the analyst enters the weights into the LIMMs system. The LIMMs system automatically decides whether the analysis passes or fails.

If a sample result changes as result of an error found through quality control the client is informed of the new result. All QC results generated by each analyst are kept in spreadsheets and the analyst's performance is tracked and control charts are generated. The lab's overall performance is also tracked in this manner.

13.4 Analytical Quality Control of TEM Air Samples

The following procedures are used to maintain a high level of quality control on TEM air sample analysis.

1. Verified Count Analysis - Verified count analysis is performed by each TEM analyst on samples with known asbestos concentrations at a rate of 1 per every

100 samples analyzed. Each analyst must maintain a 85% true positive level to remain qualified to analyze TEM samples. If an analyst falls below an 85% true positive level additional training is given until that analyst re-qualifies. The NIST 1876b is issued at least once per year for verified count analysis. The data is summarized per analyst and stored in the Quattro Pro verified count spreadsheet.

2. Replicate Analysis - Four percent of all air samples analyzed by each individual analyst are reanalyzed by another analyst. The replicate analysis is completed prior to mailing the final report.

3. Duplicate Analysis - Two percent of all air samples analyzed by each individual analyst are reanalyzed by the same analyst. The duplicate analysis is completed prior to mailing the final report.

4. Blank Analysis - A blank sample is prepared along side with each set of samples. If the results of any of the live samples within the sample set is above the clients release criteria the blank sample is analyzed to check for possible contamination. Blank samples which have a number ending in zero are also analyzed.

5. Re-preparation of filters – Three samples are randomly selected and prepared a second time for re-analysis. The preparations are compared to the original sample preparations. Particulate loading, plasma ashing texture, fiber distribution, and the overall quality of the preparations are qualitatively evaluated. The analyst records their observations in the LIMMs system, specifically in the comments section of the replicate or duplicate TEM air form.

13.5 Analytical Quality Control of TEM Water Samples

The analytical quality control for all water samples is included in the overall TEM QC program. Quality Control is performed on water samples includes the following:

1. Replicate Analysis - Four percent of all water samples analyzed by each individual analyst are reanalyzed by another analyst.

2. Duplicate Analysis - Two percent of all water samples analyzed by each individual analyst are reanalyzed by the same analyst.

3. Blank Analysis - A blank sample is prepared along side with each set of samples. If the results of any of the live samples within the sample set is above the limit of detection (LOD) the blank sample is analyzed to check for possible contamination.

4. Verified Analysis - Weekly verified analysis is performed by each TEM analyst on samples with known asbestos concentrations. Each analyst must maintain a 85% true positive level to remain qualified to analyze TEM samples. If an analyst falls below an 85% true positive level additional training is given until that analyst re-qualifies.

If a sample result changes as result of an error found through quality control the client is informed of the new result. All QC results generated by each analyst are kept in spreadsheets and the analyst's performance is tracked and control charts are generated. The lab's overall performance is also tracked in this manner.

13.6 Analytical Quality Control of TEM Settled Dust Samples

The analytical quality control for all settled dust samples is included in the overall TEM QC program. Quality Control is performed on water samples includes the following:

1. Replicate Analysis - Four percent of all settled dust samples analyzed by each individual analyst are reanalyzed by another analyst.

2. Duplicate Analysis - Two percent of all settled dust samples analyzed by each individual analyst are reanalyzed by the same analyst.

3. Blank Analysis - A blank sample is prepared along side with each set of samples. If the results of any of the live samples within the sample set is above the limit of detection (LOD) the blank sample is analyzed to check for possible contamination.

4. Verified Analysis - Weekly verified analysis is performed by each TEM analyst on samples with known asbestos concentrations. Each analyst must maintain a 85% true positive level to remain qualified to analyze TEM samples. If an analyst falls below an 85% true positive level additional training is given until that analyst re-qualifies.

If a sample result changes as result of an error found through quality control the client is informed of the new result. All QC results generated by each analyst are kept in spreadsheets and the analyst's performance is tracked and control charts are generated. The lab's overall performance is also tracked in this manner.

13.7 Interlab Quality Control

In addition in house quality control, AMA exchanges verified count samples with another NVLAP and ELAP accredited laboratory on a quarterly basis.

13.8 Data Reduction of Verified Count Samples

When all of the TEM analysts have completed the assigned sample, each individual analyst's results are compared to all other participating analyst's results. If a structure is documented by one analyst and not verified by the other analysts the grid opening is looked at again to verify the existence and identification of that structure. If there is a discrepancy between the analysts involving the identification of the mineral or the countability of a structure, the structure(s) are looked at again to resolve the discrepancy.

When all discrepancies have been resolved a total number of true positive structures and a total number of true negative structures is generated for each grid opening and for the whole sample. Each individual analyst's results are calculated using the following equations:

Percent of True Positives Correctly Identified

((Total True Positives Counted) / (Known True Positives)) X 100

Percent of True Negatives Correctly Identified

((Total True Negatives Counted) / (Known True Negatives)) X 100

Percent False Positives

((Total False Positives Counted) / (Known True Positives)) X 100

Percent False Negatives

((Total False Negatives Counted) / (Known True Positives)) X 100

All the calculated results for each verified count sample are entered into the verified count log. The results are charted and graphed to show each analyst weekly results and to show long-term results and trends.

13.9 Contamination Checks and Follow Up

Contamination checks are performed quarterly. The laboratory safety officer takes personal samples, ambient samples, and surface wipe samples for both lead and asbestos. Personal samples are collected on technicians, analysts, and support staff. Ambient air samples and surface wipe samples are collected in places where samples are handled, prepared, and stored. Samples are also collected from areas where client samples are not handled. The asbestos ambient, personal samples, and surface wipe samples are analyzed by TEM using the AHERA counting rules. The lead samples are analyzed by atomic absorption spectroscopy.

If contamination is found on any sample, including routine blank samples, an immediate investigation is initiated to determine the cause, and procedures involved in the contaminated area are suspended until the problem is resolved. The activities performed by the employee and procedures conducted in any contaminated room are thoroughly reviewed. If it is found that an employee did not follow proper procedures, the SOPs are reviewed with that person and, if necessary, additional training is given. If a procedure is found to be responsible for the contamination it is reviewed and changed to prevent future contamination. If a piece of equipment is not functioning properly, it is removed from service until it has been repaired or replaced. In all cases, the affected areas are cleaned a retested. Personal samples are taken again after any procedural change while the employee is duplicating an activity performed during the original sampling.

When it is found that there is no longer any contamination, a summary of the investigation is written detailing the original problem and corrective action.

13.10 SRM 1876b Analysis

The 1876b sample is analyzed once per year by each TEM analyst following the instructions provided by NIST. The TEM QC manager compiles the data for each analyst and the laboratory as a whole. The analysis is counted in the overall verified count percentage for the laboratory.

13.11 Intermicroscope Analysis

The difference between the two electron microscopes is checked be comparing calibration data and analysis of QC samples on both scopes. An analyst will analyze a sample with a large variety of fiber lengths on scope 1 and immediately analyze the

same sample on scope 2. Using a spreadsheet the differences in length and width of the fibers are compared and the percent difference between the scopes is calculated.

13.12 Proficiency Test Samples

When proficiency test samples arrive from any of the accrediting agencies (NVLAP, AIHA, ELAP) the samples are logged in to the database and placed on the appropriate schedule board, just as regular samples are, and given to the correct QC manager. Depending on the type of sample efforts are made to have as many of the analysts analyze the sample without damaging it. After analysis is complete the data is compiled and one analyst's results are sent for each sample. After a proficiency test is completed the remaining samples are kept indefinitely for training and as use as reference samples.

14.0 TEM TRAINING

The purpose of this section is to outline the steps needed to prepare an individual with no prior electron microscope experience to analyze TEM asbestos samples. The TEM training program is designed to prepare the individual to analyze by himself or herself. The training program shall be of a sufficient duration to ensure analyst competency and include a minimum of 2 months hands on experience conducting analysis This document outlines the basic guidelines and order followed during training. Depending on the individual or on circumstances at the time of training, the order and/or steps can be changed. A copy of the TEM training section is found in the documents section of this manual.

14.1 Sample Receiving and Labeling

The initial training given the individual is on how to properly receive and label any incoming samples. This includes:

Filling out the chain of custody.

Entering the client and sample information into the appropriate logbooks.

Posting the sample information on the appropriate scheduling board.

14.2 Sample Preparation

Training is given for both mixed cellulose ester (MCE filter) and polycarbonate filters (PC filters). Initially the individual is trained how to prepare just the filters from air, water, and settled dust. The specific training for the filtering of water and settled dust samples is given after the individual has completed TEM air training.

14.2.1 MCE Filters

Training for the preparation of MCE filters includes:

Mounting Filters onto Slides.

Plasma asher operation.

Carbon coater operation.

Filter dissolution.

The specific steps for preparing MCE filters is found in SOP 201.

14.2.2 PC Filters

Training for the preparation of PC filters includes:

Mounting Filters onto Slides.

Carbon coater operation.

Filter dissolution using a chloroform condensate washer.

The specific steps for preparing PC filters is found in SOP 202.

14.2.3 Sample and Grid Storage

As part of sample preparation training the individual is given instructions on how and where to store grids and the original samples. The individual is also trained how to properly fill out the appropriate sample storage logbooks.

14.3 Basic TEM Operation

The next phase of the individual's training covers basic electron microscope operation. Upon completion of this part of the training the individual shall be able to align the scope, obtain an electron diffraction image, take a microphotograph, and perform the weekly calibrations.

14.3.1 Alignment

Basic TEM operation begins with training the individual proper alignment of the electron microscope. Specific procedures for aligning the scope are found in SOP 301.

14.3.2 SAED and Photographic Imaging

The procedures for SAED imaging and photographing images are found in SOP 303.

14.3.3 Weekly Scope Calibrations

Certain electron microscope calibrations must be performed on a weekly basis. These include magnification calibration, spot size calibration, and camera constant calibration. The detailed instructions for performing these procedures are found in the following SOPs:

SOP 403 Camera Constant Calibration

SOP 404 Magnification Calibration

SOP 405 Spot Size Calibration

14.4 Basic EDXA Operation

This section outlines the training given for basic EDXA operation. This training is designed such that on completion the individual will be able acquire, identify, and calibrate a spectra and solve most problems that occur with EDXA. EDXA training is broken down in these categories:

EDXA generation.

Al / Cu Calibration SOP 407 & 408

Software Operation, including:
- Acquiring Spectra
- Printing Spectra
- Storing Spectra

Retrieving Spectra

Spectra Identification

Full Width Half Max. Calibration SOP 409

14.5 Asbestos Identification

This part of the training is designed to give the individual the basic skills to properly identify all the asbestos types and most of the common non-asbestos minerals routinely encountered during analysis. The NIST 1866 is used to make training grids of the common asbestos types. The NIST 1867 is used to make training grids of the uncommon asbestos types. This training begins with detailed SAED analysis followed by detailed EDXA training. Depending on the background, some individuals can complete this section quicker.

14.5.1 Electron Diffraction Training

Detailed electron diffraction training consists of the following categories:

Basic Crystallography and Mineralogy - This includes basic crystal structure, miller indices, and zone axis concepts.

Asbestos and Non-Asbestos Minerals - More detailed crystallography and elemental content of the asbestos and common non-asbestos minerals.

Single and Double Tilt Holder Operation - Instruction on how to use the X translation on the single tilt holder and the X/Y translation on the double tilt holder during SAED analysis.

Characteristic Chrysotile Pattern - Introduction to the chrysotile pattern, what causes the streaking, and the effects of the electron beam on the chrysotile pattern.

Amphibole Patterns - Introduction to the amphibole pattern, differences between chrysotile pattern and amphibole pattern, how to obtain a zone axis pattern.

SAED Pattern Indexing - Instruction on how to index chrysotile patterns and both monoclinic and orthorhombic amphibole patterns using the calculation sheets and the zone axis tables. This includes measuring distances using the loupe, measuring angles, calculating distances, and calculating angles.

14.5.2 Energy Dispersive X-Ray Analysis Training

Detailed EDXA training is given in combination with SAED training and includes the following categories:

Asbestos and Non-Asbestos Chemistry - Introduction to the elemental composition of all the asbestos minerals and the most common non-asbestos minerals encountered.

Sodium (Na) detection in crocidolite - Instruction recognizing the presence of Na in an amphibole and correct identification of crocidolite.

Mg / Si ratios on single fibril chrysotile - Instruction in the range of the Mg / Si ratio found in chrysotile and how to distinguish it from other common Mg, Si minerals such as sepiolite.

Typical EDXA problems encountered - Instruction on the limitations of EDXA analysis and typical problems routinely encountered, such as a fiber to close to the grid bar, and their solutions.

14.6 Analysis Training and Testing

When it is determined that the individual is proficient in the basic skills need to identify asbestos, analysis training commences. At completion of this training the individual shall be ready for analysis of previously analyzed samples. This training includes the following categories:

Asbestos Standards (SRM 1866 & 1867) - Review of asbestos chemistry and crystallography using the SRM 1866 and SRM 1867 asbestos standards as a reference.

Common Non-Asbestos Minerals - Review of the chemistry and crystallography of the most commonly encountered non-asbestos minerals.

Asbestos Counting Rules - Instruction and testing of the various asbestos counting protocols including: AHERA, EPA Level II, NIOSH 7402, and EPA drinking water rules.

Verified Count Training - When it is determined, through testing, that the individual has competent basic counting skills they begin analysis of verified count samples. The individual is trained how to record and map the samples.

Analysis of Previously Analyzed Samples - When the individual maintains a 85% true positive rate and remains below a 15% false negative rate they begin analysis of previously analyzed client samples.

14.7 Live Sample Analysis

After performing re-analysis and verified count analysis, and if the individual can demonstrate that they can maintain the required true positive and false negative rates, the individual begins primary analysis of live samples. At the onset 100 percent QC is performed before all samples are reported to the client. This level of QC is maintained for four weeks and/or at least 100 samples. If at the end of the four-week period the individual has maintained the required true positive and false negatives rates they begin live sample analysis with only standard QC.

Live sample analysis training also includes instruction on the following:

Paperwork (fiber count sheets, fax sheets, COC...)

Calculations

Reporting Results

Filing of Reports

Common problems associated with routine TEM analysis

14.8 Darkroom Procedures

At any point during the entire TEM program instruction in darkroom procedures can be given. Darkroom training takes approximately eight hours and includes the following:

Mixing Chemicals

Safety Procedures

Developing of Negatives

Developing Prints

Storing Negatives

Disposal of Used Chemicals

Maintaining and Cleaning the Darkroom

The purpose of this section is to describe the procedures for using the laboratory information management system, LIMS. AMA Analytical Services, Inc. uses a Microsoft Access based database, developed in house, for the LIMS. The LIMS is a secured database that utilizes different levels of user access. The LIMS is used for all aspects of client and sample management.

16.1 Security

The LIMS utilizes two different levels of user access, the Admin group and the User group. The groups allow the user different levels of access to the various forms, tables and reports within the LIMS. The network manager determines the group that a user is assigned to. The network manager also establishes a users logon name and password. Only the network manager and the laboratory manager are members of the Admin group. All other employees are members of the User group.

The LIMS program is located on the laboratory server, but can be accessed from any of the computers on the network. When accessing the LIMS the user is prompted for their logon name and password.

There is a link for the *Technical Guidelines Manual* on each computer in the laboratory. The link is password protected and only managers have access to the password. Without the password, the link can only be opened as a "read only" file.

The AMA Search Web Page, which finds all of the jobs that have been stored in the PCI .pdf file conversion database, is available only on AMA's internal computer network. The web page cannot be accessed outside of AMA's office.

16.1.1 Admin Group

The Admin group allows the user access to all aspects of the LIMS system. Members of the Admin group can delete records, change report and form formats, design new forms and reports, and write new tables, queries, code, and macros. Users with this level of access can also enter a new chain of custody, login samples, enter sample results, and access all billing and QC reports.

16.1.2 User Group

The User group allows the user restricted access of the LIMS system. Members of the User group can enter a new chain of custody, login samples, enter sample results, and access all billing and QC reports. Users with this level of access cannot access or change the design of any forms, tables, reports, or queries. They cannot delete records or change the data within certain records. If a member of the User group needs to change or delete a record they must get a member of the Admin group to do so.

16.1.3 Data Backup

The LIMS program is backed up daily to tape file that is connected to the main server. The tape is changed daily and stored off site.

Backup procedures for data generated by the AA machines is as follows: Raw analysis data is archived quarterly using the PE Data Manager. Zip files are stored on the hard drive of the instrument computer and on the L drive of the network server. Exported XLS reports are archived and backed up quarterly on the L drive. The Perkin Elmer reports folder is backed up nightly using Microsoft Backup. The Thermo SLR files (raw data and methods) are backed up onto the L drive weekly.

16.2 Client Lists and Client Prices

All client information is stored in the LIMS and can be accessed or updated using the address form. The address form is used for entering all the information for new clients. It is also used for changing any data for preexisting clients. Members of the Admin and the User group can access this form. Only members of the Admin group can delete records on this form.

Client prices are entered in the client prices form. The user must first select the client. They then select the analysis type, sample type, turnaround time, and then enter the price. Clicking on the "Add New Price" button can enter another price. Members of the Admin and the User group can access this form. Only members of the Admin group can delete records on this form.

16.3 Chain of Custody and Login of Samples

When a set of samples is submitted it is accompanied by chain of custody (COC). Each chain of custody has a unique number. The user must enter the data from the chain of custody into the LIMS. From the main screen the user clicks on the chain of custody button and selects "enter a new COC". The user then fills out all of the applicable fields on this form including: the COC number, client code, job name, job site, job number, PO number, number and type of samples, contact person, and person submitting.

After all the information is entered for the COC the samples must be logged in to the LIMS. From the COC form that has just been filled out the user clicks on the "login samples button". Each sample is assigned a unique laboratory number. The user then types in or selects the COC number from the combo box list in the COC box. The user must enter in the client sample number, analysis type, sample type, and select the turnaround time from the appropriate combo box. The price is automatically set based on the information that has just been entered. The laboratory number is automatically assigned for each sample that is entered. To add another sample the user clicks on the "add a new sample" button. The next sequential laboratory number is automatically assigned to the next sample.

Members of the User and the Admin group can fill out the chain of custody for and the login form.

(See SOP 101 in Appendix A of this Manual)

16.4 Billing and Invoicing

When a set of samples has been completed it is invoiced. From the main screen the user clicks on the chain of custody form and selects "work with an existing COC". The user enters the COC number they wish to work with. To invoice the COC the user clicks on the invoice check box. This automatically sets the date invoiced and assigns the sequential invoice number to the COC. Each invoice number is unique and can only be used once. To print the invoice the user clicks on the "preview invoice" button. The user is prompted "Is

the invoice box checked". If yes, a preview of the invoice is displayed and the user can print the invoice.

16.4.1 Billing Summary Reports

The LIMS system displays summaries of the billing in many ways. To access these reports the user clicks on the "billing" button from the main screen. The billing screen allows the user to view reports on invoiced projects, projects not yet invoiced, monthly and annual billing summaries, monthly and annual sample volumes, average sample prices, and many other billing and volume summaries.

16.5 Entering Sample Results and Viewing Reports

The results for every type of analysis performed by AMA Analytical Services, Inc. are entered into the LIMS. From the main screen the user must open the correct COC record on the COC form and check the analyzed box. This will automatically set the date analyzed to the current date. The user then returns to the main screen. From the main screen the user clicks on the PCM, PLM, TEM, or Metals button. This will open the menu form each of the types of analysis. From the analysis menus the user selects the type of samples they are working with.

16.5.1 PCM Analysis

From the PCM menu the user clicks on the PCM analysis button and selects enter new results. This opens the form for PCM data entry. The user selects the COC they wish to enter results for from the COC combo box. The user must then select the sample number they want enter results for from the sample number combo box. The user then fills in all applicable data fields including: analyst ID, sample type, filter size, volume, total fibers, number of fields, and blank count. To add another result the user clicks on the "add new result" button and selects the next sample from the sample number combo box. The COC, analyst ID, and filter size are automatically the same as the previous result but can be changed if necessary. When all the sample results have been entered the user can preview the report by clicking the "Preview Report". The user will be prompted to check the analyzed box on the COC form if they have not already done so. The user can check the accuracy of the results on the screen. If everything is correct the user can then print the report.

16.5.2 PLM EPA 93 Results

From the PLM main menu the user clicks on the PLM results button and selects enter new results. The user selects the COC they wish to enter results for from the COC combo box. The user must then select the sample number they want enter results for from the sample number combo box. The user then fills in all applicable data fields including: analyst ID, all of the applicable percentage fields, and sample color. If the sample was not analyzed but needs to appear on the report the user clicks the "sample not analyzed" check box. For samples recommended for TEM check the appropriate check box. To add another result the user clicks on the "add new result" button and selects the next sample from the sample number combo box. When all the sample results have been entered the user can preview the report by clicking the "Preview Report". The user will be prompted to check the analyzed box on the COC form if they have not already done so. The user can check the accuracy of the results on the screen. If everything is correct the user can then print the report.

16.5.3 PLM Point Count Results

From the PLM main menu the user clicks on the PLM results button and selects enter new results. The user selects the COC they wish to enter results for from the COC combo box. The user must then select the sample number they want enter results for from the sample number combo box. The user then fills in all applicable data fields including: analyst ID, all of the applicable point and percentage fields, and sample color. If a stratified point count was performed and the scanning negative option (SNO) was used the user must check the SNO box. If the sample was not analyzed but needs to appear on the report the user clicks the "sample not analyzed" check box. For samples recommended for TEM check the appropriate check box. To add another result the user clicks on the "add new result" button and selects the next sample from the sample number combo box. When all the sample results have been entered the user can preview the report by clicking the "Preview Report". The user will be prompted to check the analyzed box on the COC form if they have not already done so. The user can check the accuracy of the results on the screen. If everything is correct the user can then print the report.

16.5.4 TEM Air Results

From the TEM main menu the user clicks on the TEM Air results button and selects enter new results. The user selects the COC they wish to enter results for from the COC combo box. The user must then select the sample number they want enter results for from the sample number combo box. The user then fills in all applicable data fields including: analyst ID, sample type, grid box number, row number, filter type, filter size, porosity, volume, area analyzed, asbestos types and amounts, and number of non-asbestos structures. To add another result the user clicks on the "add new result" button and selects the next sample from the sample number combo box. Many of the fields will be automatically filled with the same data as the previous sample but can be changed if necessary. When all the sample results have been entered the user can preview the report by clicking one of the "Preview Report" buttons. This depends on the type of TEM air analysis being done: AHERA, AHERA style, NIOSH 7402, or EPA Level 2. The user will be prompted to check the analyzed box on the COC form if they have not already done so. The user can check the accuracy of the results on the screen. If everything is correct the user can then print the report.

16.5.5 NOB Results

From the TEM main menu the user clicks on the NOB results button and selects enter new results. The user selects the COC they wish to enter results for from the COC combo box. The user must then select the sample number they want enter results for from the sample number combo box. The user then fills in all applicable data fields including: analyst ID, sample type, grid box number, row number, vial weight, vial and sample weight, post ash weight, gross filter weight, and the estimated asbestos percentages. To add another result the user clicks on the "add new result" button and selects the next sample from the sample number combo box. Many of the fields will be automatically filled with the same data as the previous sample but can be changed if necessary. When all the sample results have been entered the user can preview the report by clicking one of the "Preview Report" buttons. This depends on type of sample and whether the client submitted the weight data for a residue. The user will be prompted to check the analyzed box on the COC form if they have not already done so. The user can check the accuracy of the results on the screen. If everything is correct the user can then print the report.

16.5.6 TEM Water Results

From the TEM main menu the user clicks on the NOB results button and selects enter new results. The user selects the COC they wish to enter results for from the COC combo box. The user must then select the sample number they want enter results for from the sample number combo box. The user then fills in all applicable data fields including: analyst ID, sample type, aliquot, EFCA, area analyzed, total fibers, total long fibers, and asbestos types. To add another result the user clicks on the "add new result" button and selects the next sample from the sample number combo box. Many of the fields will be automatically filled with the same data as the previous sample but can be changed if necessary. When all the sample results have been entered the user can preview the report by clicking the "Preview Report". The user will be prompted to check the analyzed box on the COC form if they have not already done so. The user can check the accuracy of the results on the screen. If everything is correct the user can then print the report.

16.5.7 TEM Settled Dust

From the TEM main menu the user clicks on the NOB results button and selects enter new results. The user selects the COC they wish to enter results for from the COC combo box. The user must then select the sample number they want enter results for from the sample number combo box. The user then fills in all applicable data fields including: analyst ID, surface area sampled, EFCA, volume filtered, area analyzed, and asbestos types and amounts. If the sample is qualitative the user only needs to check the asbestos present box. To add another result the user clicks on the "add new result" button and selects the next sample from the sample number combo box. Many of the fields will be automatically filled with the same data as the previous sample but can be changed if necessary. When all the sample results have been entered the user can preview the report by clicking one of the "Preview Report" buttons. This depends on type of sample, quantitative or qualitative. The user will be prompted to check the analyzed box on the COC form if they have not already done so. The user can check the accuracy of the results on the screen. If everything is correct the user can then print the report.

16.5.8 Lead Results

Before entering lead results the user must enter their QC data as described in section 16.6.6. From the lead main menu the user clicks on the Lead results button and selects enter new results. The user selects the COC they wish to enter results for from the COC combo box. The user must then select the sample number they want enter results for from the sample number combo box. The user then fills in all applicable data fields including: analyst ID, prepared ID, checked ID, SDG number, analysis type, sample type, slope, intercept, absorbance, dilution factor, dry sample weight, area wiped, and air volume. Depending on the type of sample analyzed some of these fields may not be required. To add another result the user clicks on the "add new result" button and selects the next sample from the sample number combo box. Many of the fields will be automatically filled with the same data as the previous sample but can be changed if necessary. When all the sample results have been entered the user can preview the report by clicking one of the "Preview Report" buttons: lead, copper, or TCLP. The user will be prompted to check the analyzed box on the COC form if they have not already done so. The user can check the accuracy of the results on the screen. If everything is correct the user can then print the report.

16.6 Quality Control

The LIMS is used for tracking many of the QC requirements for PCM, PLM, TEM, and Lead. From the main screen the user selects the type of analysis they wish to work. From each analysis type main menu the user can select the type of QC needed.

16.6.1 PCM Quality Control

To enter a reference result the user clicks on the "enter reference result" button on the PCM menu and fills out all the applicable fields. The LIMS will automatically pass the sample if the result is within the upper and lower limit range. If the result is outside the acceptable range the sample will fail.

To enter a duplicate QC result the user will enter the QC data in the appropriate fields on the PCM results screen. This is done at the same time as the user enters their original results. The LIMS compares the original result to the QC result and indicates whether the QC result passes or fails.

The LIMS system summarizes all the PCM QC. To view these reports the user clicks on the "PCM QC" button from the PCM menu. There are reports on the lab's overall duplicate performance, individual analysts duplicate performance, overall lab's reference sample results, and the individual analysts performance. There is also report comparing the number of PCM samples analyzed to the number of QC analysis performed. All of the reports prompt the user for a start and finish date.

16.6.2 PLM Quality Control

To enter a duplicate QC result the user will enter the QC data in the appropriate fields on the PLM results screen. This is done at the same time as the user enters their original results.

Replicate results are entered in the LIMS by the user in groups. To enter a set of replicate results the user clicks on the PLM results button and selects work with existing records. The user enters the COC number of the replicate result they need to enter. The user fills out all of the applicable replicate QC fields. To add another replicate QC result the user clicks on the "find an AMA number" button and enters the AMA sample number needed.

The LIMS system summarizes all the PLM QC. To view these reports the user clicks on the "PLM QC" button from the PLM menu. There are reports on the lab's overall replicate performance, individual analyst's replicate performance, overall lab's duplicate sample results, and the individual analyst's duplicate sample results. There is also report comparing the number of PLM samples analyzed to the number of QC analysis performed. All of the reports prompt the user for a start and finish date.

16.6.3 TEM Air Quality Control

The LIMS system is used to assign replicate and duplicate QC samples monthly. At the beginning of each month the TEM QC manager assigns the QC. The user assigns the QC by clicking on the "Summaries" button on the main menu. The user selects the "Total TEM Airs by Date and Analyst" button. The user fills in the start and finish date fields and selects TEM analysts. By clicking on the Replicate QC button or the Duplicate QC button the LIMS will randomly select the appropriate number of

replicate or duplicate QC samples. The amount of samples selected is based on that analyst's quantity of samples analyzed in the specified time period.

To enter a duplicate QC result or a replicate QC result the user will enter the QC data in the appropriate fields on the TEM results screen. Replicate and duplicate QC results are usually entered in groups. To add another replicate or duplicate QC result the user clicks on the "find an AMA number" button and enters the AMA sample number needed.

The LIMS system summarizes all the TEM air QC. To view these reports the user clicks on the "TEM air QC" button from the TEM menu. There are reports on the lab's overall replicate performance, individual analyst's replicate performance, overall lab's duplicate sample results, and the individual analyst's duplicate sample results. There is also report comparing the number of TEM air samples analyzed to the number of QC analysis performed. All of the reports prompt the user for a start and finish date.

16.6.4 NOB Quality Control

The LIMS system is used to assign replicate and duplicate QC samples monthly. At the beginning of each month the TEM QC manager assigns the QC. The user assigns the QC by clicking on the "Summaries" button on the main menu. The user selects the "Total NOBs by Date and Analyst" button. The user fills in the start and finish date fields and selects a NOB analyst. By clicking on the Replicate QC button or the Duplicate QC button the LIMS will randomly select the appropriate number of replicate or duplicate QC samples. The amount of samples selected is based on that analyst's quantity of samples analyzed in the specified time period.

To enter a PLM and/or TEM duplicate QC result or a replicate QC result the user will enter the QC data in the appropriate fields on the NOB results screen. Replicate and duplicate QC results are usually entered in groups. To add another replicate or duplicate QC result the user clicks on the "find an AMA number" button and enters the AMA sample number needed.

The LIMS system summarizes all the NOB QC. To view these reports the user clicks on the "NOB QC" button from the TEM menu. There are reports on the lab's overall replicate performance, individual analyst's replicate performance, overall lab's duplicate sample results, and the individual analyst's duplicate sample results. There is also report comparing the number of TEM air samples analyzed to the number of QC analysis performed. All of the reports prompt the user for a start and finish date.

16.6.5 Settled Dust and Water Quality Control

The TEM QC manager assigns settled dust and water sample QC on a as needed basis. Compared to the other types of TEM samples the frequency of these types of analysis is much less. From the summary screen the LIMS is used to list each one of these types of analysis each TEM analysts has performed. The TEM QC manager randomly selects QC samples as needed.

16.6.6 Lead Quality Control

For each set of sample the user must enter in QC data into the LIMS. From the main menu the user clicks on the "Lead Results" button and selects the type of lead QC they wish to enter: Lead Flame, Lead Furnace, or Lead Water. The user selects

"Enter New SDG" and enters all applicable data into the fields. Some of the fields will be automatically filled but can be changed if necessary. The user then enters their lead results in by clicking on the "Lead Results" button and selecting "enter new results. The results are entered in as described in section 16.5.8. The user prints the QC report and checks the data making all necessary corrections. The user compares the results on the QC report to those on the Lead report. If everything checks the QC report and the Lead report are printed.

End of Manual

SOP No. 409

TITLE: **PROCEDURE FOR FULL WIDTH, HALF MAX DETERMINATION**

Approved & Authorized: _____

G. Edward Carney, Technical Manager

Approved & Authorized: _____

Andreas Saldivar, Laboratory Director

Approved & Authorized: _____

Joseph Coco, QA Manager

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 409

TITLE: **PROCEDURE FOR FULL WIDTH, HALF MAX DETERMINATION**

Sign: _____ Date: 6/24/10

Print Name: Michael Craig

Sign: _____ Date: 6/23/10

Print Name: G E Carney

Sign: _____ Date: 6/24/10

Print Name: Andres Saldivar

Sign: _____ Date: 6/24/10

Print Name: Ang Cao

Sign: _____ Date: _____

Print Name: _____

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 409 Procedure for Full Width, Half Max Determination

Sign: Michael Greenberg Date: 10/30/12

Print Name: Michael Greenberg

Sign: Robert Privette Date: 10/30/12

Print Name: ROBERT PRIVETTE

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 409 Procedure for Full Width, Half Max Determination

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Thermo Noran Systes

The Thermo Noran system will automatically calculate the resolution.

Insert the manganese specimen and focus on a particle towards the center of a grid opening. Choosing a grid opening near the center of the grid works best.

Begin acquiring a spectrum and adjust the count rate to 3,000 or below.

Stop acquiring.

Bring up the expert item on the menu bar by holding the ctrl, shift, and alt buttons while simultaneously clicking on the toolbar.

From the expert menu select "Measure FWHM"

Select element "Mn" and Line "K"

Select "Peak Counts" and set the maximum to 2000.

Set the "No. Trials" to 5.

Click on run.

The software will begin acquiring the first spectrum and will continue acquiring until the fifth spectrum is done. During this process monitor the count rate and adjust the particle if it becomes to high.

When the process is complete record the value for each spectra in the database and save the file.

END SOP 409

SOP No. 408

TITLE: **EDXA CALIBRATION: THERMO-NORAN**

Approved & Authorized: _____

G. Edward Carney, Technical Manager

Approved & Authorized: _____

Andreas Saldivar, Laboratory Director

Approved & Authorized: _____

Joseph Coco, QA Manager

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

Sign: _____ Date: 6/22/10

Print Name: _____

Sign: _____ Date: 6/23/10

Print Name: G E Carney

Sign: _____ Date: 6/24/10

Print Name: Andreas Saldiver

Sign: _____ Date: 6/24/10

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 408 EDXA Calibration: Thermo-Noran Analyzer

Sign: Michael Greenberg Date: 10/30/12

Print Name: Michael Greenberg

Sign: Robert Privette Date: 10/30/12

Print Name: ROBERT PRIVETTE

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 408 EDXA Calibration: Thermo-Noran Analyzer

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

1. Insert aluminum-copper grid into microscope.

2. Adjust to eucentric position with "Z" control and focus.

3. Tilt specimen along X-axis by moving control in a clockwise direction to 45 degrees.

13. Set microscope to spot-size four. The condenser aperture will be adjusted depending on the count rate.

14. Double click on the Spectra Plus icon.

15. Place the beam spot anywhere on the grid opening or on a particle.

7. Click on go.

8. If the count rate is above 5000 go to a higher numbered condenser aperture and click stop.

9. Click on the acquisition menu and select auto calibration.

10. You are prompted to move the cursor to the peak that you want to calibrate with, typically the copper peak.

11. Select "No" when asked if you want to save the spectra.

12. When the auto calibration menu is displayed select Cu and click start.

13. When the calibration is complete, note it in the logbook as "Cu ok" if you calibrated with copper. If you calibrated with another element, note it.

END SOP 408

SOP No. 406

TITLE: **BEAM DOSE CALIBRATION PROCEDURE**

Approved & Authorized: _____

G. Edward Carney, Technical Manager

Approved & Authorized: _____

Andreas Saldivar, Laboratory Director

Approved & Authorized: _____

Joseph Coco, QA Manager

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 406

TITLE: **BEAM DOSE CALIBRATION PROCEDURE**

Sign: _____ Date: 6/22/10

Print Name: _____

Sign: _____ Date: 6/23/10

Print Name: GE Carney

Sign: _____ Date: 6/24/10

Print Name: Andreas Soldiver

Sign: _____ Date: 6/24/10

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 406 Beam Dose Calibration Procedure

Sign: Michael Greenberg Date: 10/30/12

Print Name: Michael Greenberg

Sign: Robert Privette Date: 10/30/12

Print Name: ROBERT PRIVETTE

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 406 Beam Dose Calibration Procedure

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

To determine the damage to a fiber by an electron beam, obtain a focused image of a chrysotile (most easily damaged) fiber on the phosphor screen.

Observe the fiber's diffraction pattern for 15 seconds under normal diffraction parameters.

Photograph the diffraction pattern and check for beam damage.

If damage is noted, adjust the position of the illuminance-controlling knobs so that fewer electrons per unit area bombard the specimen, and repeat the procedure, using a fresh fiber, until no damage is noted. Beam dose damage for all future analysis can be checked during reanalysis of samples and verified counting during QC monitoring.

Frequency of beam dose calibration will be quarterly, or as required following QC monitoring.

NVLAP requires that a chrysotile fiber be stable in the electron beam for 15 seconds each without inducing beam damage.

END SOP 406

SOP No. 405

TITLE: SPOT SIZE MEASUREMENT PROTOCOL

Approved & Authorized: _____

G. Edward Carney, Technical Manager

Approved & Authorized: _____

Andreas Saldivar, Laboratory Director

Approved & Authorized: _____

Joseph Coco, QA Manager

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 405

TITLE: **SPOT SIZE MEASUREMENT PROTOCOL**

Sign: _____ Date: 6/22/10

Print Name: Michael Cough

Sign: _____ Date: 6/23/10

Print Name: G E Carney

Sign: _____ Date: 6/23/10

Print Name: Andreas Saldivar

Sign: _____ Date: 6/24/10

Print Name: Ang Cao

Sign: _____ Date: _____

Print Name: _____

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 405 Spot Size Measurement Protocol

Sign: Michael Greenberg Date: 10/30/12

Print Name: Michael Greenberg

Sign: Robert Privette Date: 10/30/12

Print Name: ROBERT PRIVETTE

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 405 Spot Size Measurement Protocol

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

AHERA mandates that the TEM used for asbestos analysis be able to produce a spot size at crossover of 250nm or less.

AMA Analytical Services, Inc. operates JEOL electron microscopes whose C1 (spot size) controls are regulated by click-stop potentiometers.

Photographs are taken at crossover of astigmatic beams at spot sizes 2 and 3 on a weekly basis. To obtain the photographic negative with the least density (and the most accurate diameter), exposure time of 0.12 seconds or 0.35 seconds is used. The normal 150-micron condenser aperture is used. No objective aperture is used, since it is not used during EDXA analysis.

To determine the spot size measurement, the negative is placed on a light box and 4 diameters are measured using a scaled loupe with 0.1 mm increments.

Enter the values obtained in the Quattro Pro spreadsheet. Make sure that you record the values in the correct table for scope 1 or scope 2.

END SOP 405

SOP No. 404

TITLE: **TEM MAGNIFICATION CALIBRATION PROCEDURE**

Approved & Authorized: _____

G. Edward Carney, Technical Manager

Approved & Authorized: _____

Andreas Saldivar, Laboratory Director

Approved & Authorized: _____

Joseph Coco, QA Manager

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No: 404

TITLE: **TEM MAGNIFICATION CALIBRATION PROCEDURE**

Sign: _____ Date: 6/22/10

Print Name: _____

Sign: _____ Date: 6/23/10

Print Name: G E Carney

Sign: _____ Date: 6/24/10

Print Name: Andreas Saldivar

Sign: _____ Date: 6/24/10

Print Name: Amy Cao

Sign: _____ Date: _____

Print Name: _____

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 404 TEM Magnification Calibration Procedure

Sign: Michael Greenberg Date: 10/30/12

Print Name: Michael Greenberg

Sign: Robert Privette Date: 10/30/12

Print Name: ROBERT PRIVETTE

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 404 TEM Magnification Calibration Procedure

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

This procedure is performed weekly or after physical or electronic adjustment of the microscope. A magnification standard (E.F.Fullam #10021) of 2160 intervals/mm is inserted at the eucentric point of the TEM specimen stage and focused.

SCREEN MAGNIFICATION:

The number of grating lines lying across the large (74mm) scribed circle is counted; fractions are estimated.

To calculate the screen magnification, insert the # of lines counted into the formula:

M=XG/Y Where: X = the distance, in mm, between grating lines (the diameter of the circle, or 74mm), Y = the number of counted grating intervals observed across the diameter, and G = the number of grating intervals per mm (2160).

Example: X = 74mm G = 2160 intervals/mm Y = 10.4 intervals

Answer: M = 15,370 times magnification

NEGATIVE (FILM PLANE) MAGNIFICATION:

To determine the magnification of the TEM at the film plane, photograph the same image that was measured above. Measure, on the resulting negative, the distance (in mm) between any 2 interval boundaries; the more intervals measured, the smaller the measuring error.

Using the same formula as above, X = the distance across the measured boundaries, in mm Y = an integer equal to the number of intervals traversed in "X".

Example: X = 35.5mm Y = 4 intervals G = 2160 intervals/mm

Answer: M = 19,170 times magnification.

Repeat the procedure for 14,000x and 2,900x.

Enter the values in the Quattro Pro spreadsheet. Make sure that you record the values in the correct table for scope 1 or scope 2.

END SOP 404

SOP NO. 403

TITLE: **PROCEDURE FOR OBTAINING THE CAMERA CONSTANT**

Approved & Authorized: _____

G. Edward Carney, Technical Manager

Approved & Authorized: _____

Andreas Saldivar, Laboratory Director

Approved & Authorized: _____

Joseph Coco, QA Manager

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP NO. 403

TITLE: **PROCEDURE FOR OBTAINING THE CAMERA CONSTANT**

Sign: _____ Date: 6/22/10

Print Name: Michael Creglia

Sign: _____ Date: 6/23/10

Print Name: GECarney

Sign: _____ Date: 6/24/10

Print Name: Andreas Saldivar

Sign: _____ Date: 6/24/10

Print Name: Amy Cao

Sign: _____ Date: _____

Print Name: _____

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 403 Procedure for Obtaining the Camera Constant

Sign: Michael Greenberg Date: 10/30/12

Print Name: Michael Greenberg

Sign: Robert Privette Date: 10/30/12

Print Name: ROBERT PRIVETTE

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 403 Procedure for Obtaining the Camera Constant

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Photo Generation

Gold standards for camera constant preparation are available commercially (Ted Pella, Inc. #10092). These can be manufactured in the lab by placing a torn prep on its grid, on a glass slide. Grids containing no sample, but coated with a thin FormVar (polyvinyl formal) film may also be used. Affix the grid to the slide with tape. Place in the sputter coater and coat the slide with gold for 20 seconds at 35 milliamps. Alternatively, a 4mm length of fine (0.2mm) gold wire can be wrapped around a tungsten basket and evaporated in the Evaporative Coater unit (see SOP for Carbon Coating of specimens).

The calibration of the camera constant is a once-weekly procedure, but may easily be performed at any time, and must be performed after any service or dislocation of or to the lenses of the microscope.

Insert the standard into the appropriate specimen holder and then into the column.

At 19,000x (display magnification), adjust the specimen holder so that the specimen is at the eccentric position. (refer to JEOL Manual for details) If using a double tilt holder, be sure that the X tilt has been adjusted to 0 before any other adjustments to other axes are made.

Raise the phosphor screen using control button on right side of column.

Spread the beam and focus the gold film.

Enter SA-MAG mode at 19,000-display magnification and focus the film again.

Note the Objective Lens current on the daily check-out log.

Insert the Selected Area Aperture to the smallest opening and center it.

Focus the edge of the aperture with the SA-MAG focus knob.

Enter the SA-DIFF mode at 22cm camera length.

Spread the beam to a comfortable level.

Adjust the diffraction focus knob so that the smallest central spot is obtained.

Place the beam-stop so that it blocks the central beam.

Switch to 55cm or 83cm camera length as appropriate.

Insert objective aperture 2 on scope 2 and aperture 3 on scope 1. ***See SOP 410 for more details on objective aperture calibration.***

Move the objective aperture away from the central ring to the outer rings.

Spread the coarse C2 control fully clockwise, then back it off 1 click in the counter-clockwise direction.(Potentiometers are not meant to be used in either of their fully-traversed positions due to inherent electrical noise problems.)

Spread the fine C2 control fully clockwise, then back off 1/10 of a turn.

Advance one sheet of film using film advance button.
Press the MANUAL exposure setting button.

Adjust the exposure time to 45 seconds.

Unless working in total darkness, cover the viewing port of the microscope to prevent ambient light from striking the film during exposure.

Press the "Expose" button.

When the red "exposing" light goes out, press and hold the film advance button. This will allow another exposure to be made on the same film plate.

Remove the objective aperture.

Expose the film for another 45 seconds, be sure to press and hold the film advance button again at the end of this exposure.

Remove the beam-stop, carefully. DO NOT DISTURB THE SPECIMEN STAGE CONTROLS.

Reset the exposure time to 0.12 seconds.

Press the "Expose" button again.

Allow the exposure to continue and the film to be "moved" into the exposed film collection container.

Process the film in the usual manner for TEM film (see TEM Photograph Instructions), and dry it completely before continuing.

MANUAL PROCEDURE FOR MEASURING THE CAMERA CONSTANT

For manual calculation of the camera constant, use the diffraction pattern worksheets for calculating the camera constant.

Enter the negative number, camera length, analyst name, and date in the spaces provided.

Measure 8 equally spaced radii and enter their lengths in the spaces provided.

Use a 4x Magnifier/loupe (SPI Supplies #2251-AB) for measuring. Place the loupe so that the calibrated line passes through the center beam spot at the zero mm point. Then read, in millimeters, the distance from the center spot to each perimeter of the innermost gold ring and write down each of these two values in two of the spaces provided on the form (r1=_____ and r2=_____, for example).If you detect appreciable width of the ring itself, measure as accurately as possible to the middle of the actual ring.

Rotate the loupe 45° and read radii again, until you have measured at least 4 radii. Compute their average and fill in the appropriate space, provided. AMA Analytical Services uses a gold (Au) sample as our standard; multiply the average radius value by 2.355 Angstroms to compute the camera constant. The constant is calculated in the units "millimeter Angstroms". The millimeters will drop out when calculating the actual d-spacing of an unknown diffraction pattern.

CRYSTAL WORKSHEET

Negative #_____
Camera Length:_____cm
Analyst:_____
Date:_____

r1= _____mm
r2= _____mm
r3= _____mm
r4= _____mm

r5= _____mm
r6= _____mm
r7= _____mm
r8= _____mm

Average=_____mm
d-Spacing for Au= x2.355 Angstroms
Camera Constant = _____ mm Angstroms

END SOP 403

SOP No. 401

TITLE: **PROCEDURE FOR MEASURING GRID OPENINGS**

Through the Phase Contrast Microscope

Approved & Authorized: _____

G. Edward Carney, Technical Manager

Approved & Authorized: _____

Andreas Saldivar, Laboratory Director

Approved & Authorized: _____

Joseph Coco, QA Manager

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 401

TITLE: **PROCEDURE FOR MEASURING GRID OPENINGS**

Through the Phase Contrast Microscope

Sign: Chon Simpha Date: 6-22-10

Print Name: CHON SIMPHA

Sign: Date: 6/22/10

Print Name: Michael Grayden

Sign: Date: 6/23/10

Print Name: GE Carney

Sign: Date: 6/24/10

Print Name: Andreas Saldivar

Sign: Date: 6/24/10

Print Name: Amy Cen

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 401 Procedure for Measuring Grid Openings through the Phase Contrast Microscope

Sign: _____ Date: Oct 30, 2012

Print Name: ROBERT PRIVETTE
Bob

Sign: _____ Date: 10/31/12

Print Name: KRISHA ORDANIEL
Krisha

Sign: _____ Date: 10/30/12

Print Name: Michael Greenberg
Mike G

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 401 Procedure for Measuring Grid Openings through the Phase Contrast Microscope

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Commercially available 200-mesh, indexed, copper grids are used during the sample preparation steps.

To determine accurately the air concentration of asbestos, the grid opening area must be determined prior to sample preparation.

The procedure for determining the grid opening (GO) area is as follows:

10 vials containing 100 grids/vial is defined as one lot. The lot is labeled in accordance with the purchase order number of the lot.

2 grids from each vial are randomly selected for measurements.

20 Gos from each grid are measured along the X axis and 20 Gos from each grid are measured along the Y axis.

Measurement data, in mm, are entered onto the GO calibration worksheet. The measurements are made using a phase-contrast microscope equipped with a calibrated Walton-Beckett graticule. The Walton Beckett graticule is calibrated using a commercially available stage micrometer (Olympus Corp. or Nikon, e.g.).

DATA CRITERIA:

The average GO area, in mm^2, for each analyzed grid is calculated.

The average GO area in mm^2 for each vial is calculated by obtaining the average of the two grids' average measurements.

The average GO area in mm^2 for the lot is calculated by averaging the average GO value for each of the 10 vials.

Grid opening calibration worksheets are maintained by the TEM Laboratory Supervisor.

ACCEPTANCE CRITERIA:

If an individual grid measurement falls outside of the accepted range ($\pm$ 5% of the calculated average for that lot), then the vial from which that grid was taken will not be included in the lot.

END SOP 401

TITLE: PHOTO PROCESSING, DARK ROOM PROCEDURES

Approved & Authorized: _____

G. Edward Carney, Technical Manager

Approved & Authorized: _____

Andreas Saldivar, Laboratory Director

Approved & Authorized: _____

Joseph Coco, QA Manager

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 305

TITLE: **PHOTO PROCESSING, DARK ROOM PROCEDURES**

Sign: _____ Date: 6/10/10

Print Name: Michael Creighton

Sign: _____ Date: 6/23/10

Print Name: G E Carney

Sign: _____ Date: 6/24/10

Print Name: Andreas Saldivar

Sign: _____ Date: 6/24/10

Print Name: Ang Cao

Sign: _____ Date: _____

Print Name: _____

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 305 Photo Processing, Dark Room Procedures

Sign: Michael Greenberg Date: 10/30/12

Print Name: Michael Greenberg

Sign: Robert Prwette Date: 10/30/12

Print Name: ROBERT PRWETTE

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 305 Photo Processing, Dark Room Procedures

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

To prepare the reagents:

Use Kodak D-19 film developer to develop the films. Purchase this chemical in powdered form to make one gallon (3.8 Liters). Wearing protective clothing and eyewear. In a fume extraction cabinet or well ventilated area, place 3 quarts (2.85 Liters) of clean, warm temperature water into a clean, brown, gallon jug. Carefully pour the contents of the D-19 envelope into the jug, using a funnel. Cap the jug and agitate gently to dissolve the powder. A stir-plate will facilitate the dissolution process. When fully dissolved, raise the level of the liquid, using water, to the 1-gallon (3.8 Liter) level, cap the jug and agitate gently to thoroughly mix. Mark this container clearly with a label stating "STOCK D-19 FILM DEVELOPER SOLUTION. TO USE: DILUTE 1 PART D-19 WITH 2 PARTS WATER. DO NOT PROCESS FILM WITH UNDILUTED D-19!"

In a second, brown, 3.8 Liter jug, pour 2.533 Liters of clean, room temperature, water. To this, add 1.266 Liters) of STOCK D-19 Film Developer solution. Cap the jug and mix well. Mark this container clearly with a label stating: "WORKING D-19 FILM DEVELOPER SOLUTION. THIS D-19 IS ALREADY DILUTED AND READY FOR USE!" Diluted D-19 will be discarded after each day's use.

Use Kodak Fixer* or Rapid Fixer (for films and papers) to stop the development of and permanently preserve the film. Purchase Rapid Fixer chemical in concentration liquid consisting of Part A and Part B. Wearing protective clothing and eye wear. In a fume extraction hood or well ventilated area, place 3 quarts (2.85 Liters) of clean, room temperature water into a clean, brown 1-gallon jug. Into the water, carefully pour the contents of the Fixer packet. When fully dissolved, raise the level of the liquid, using water, to the 1-gallon (3.8 Liter) level, cap the jug and agitate gently to thoroughly mix. Mark this container clearly with a label stating: "WORKING FIXER. THIS FIXER IS READY FOR USE. DO NOT DILUTE"

*Fixer dust is very fine and easily inhaled. Be sure to mix this and all chemicals in an appropriate, safe environment, i.e. a chemical fume hood. Never mix Fixer in the room that houses the TEM; tiny crystals of Fixer can invade the column and settle on the film, acting as an occluding dust particle to mar the image. In addition, the Fixer particle, if it adheres to the film, will prevent the film from being developed properly, leaving a small, clear hole on the processed negative. This will then produce a small black spot on the resulting positive print image.

On a prominently posted chart, record the date that each bottle was filled. Change the Working solutions after 120 TEM films have been processed. Change the Stock D-19 when it turns a medium brown in color or after 3 months, which ever comes first.

To process negatives:

The laboratory has its own tank-development system. Ensure that it has been set up properly with containers filled with Working D- 19 developer, rinse water (running), and Working Fixer.

Wash your hands of all grease and thoroughly dry them. Don clean, lint-free gloves.

Turn on the safelight(s) (OC or sodium vapor safelights) in the darkroom and turn off all other lights. Cover any light leaks that emanate from wall/ceiling joints, ceiling tiles, doorjambs and thresholds, or ready lights of functioning equipment, then return to the TEM room. Have an empty exposed film receiver box at hand.

Under safelight conditions, or very dim light:

Turn the silver colored handle of the TEM camera housing fully clockwise. Pneumatic valves will function and air will be admitted into the camera chamber. Until the camera door has been re-closed and the camera pumped down to high vacuum, the JEOL 100CX-II will not allow you to generate a beam.

When a vacuum no longer pulls the camera door against its gasket, gently pull the door open.

Grasp the stainless steel horizontal handle of the camera and pull it completely out, toward you; it will not fall out.

The stainless steel box immediately in front of you contains the exposed films. The box behind it (closer to the column) contains unexposed films. The exposed film canister (receiving canister) is always the one immediately in front of you (farthest from the column).

Remove both boxes, remembering which box contains which type of film. TEM film can be ruined by room light: Do not allow the sliding lids of either canister to open.

Replace the used canisters with a full canister of unexposed film (preferably from the scope dessicator) and an empty receiving canister, return the camera assembly into the TEM and close and lock the camera door.

Enter the darkroom, turn on the warning light above the outside of the darkroom door and close the door securely behind you.

Open the canister of exposed films and, one at a time, carefully remove each negative from its cassette and place it, vertically, into a pair of parallel grooves of a Plexiglas development rack. Handle the films by their edges only. If you must touch a surface of any film, do so along the long sides, only, up to 4mm in from any long edge. The racks currently employed will hold 24 sheets of film. Be sure that each sheet of film is gently bowed in the same direction. Usually this means that the emulsion (light) side is on the inside (concave) face.

If you have more than one rack's worth, continue to fill the grooves of additional racks that you may have until you run out of film or racks.

Place the first rack of film into a running bath of 68-degree (F) water. In practice, the actual temperature of the water is not critical for our work, but should be in the range of 60 to 75 degrees F. After 30 seconds, remove the first rack from the water bath and allow it to drain for 5 seconds. Tilt the rack to one side for more complete drainage.

Place the rack into Working D-19 developer solution (at 68-degrees F.) and start the timer for 4 minutes. For the first 15 seconds, "agitate" the film by repeatedly raising and lowering the rack in rapid succession. This action will dislodge any air bubbles that could lead to uneven development of the film.

Every 30 seconds, agitate the film in a similar manner for 5 seconds.

When 5 seconds are left on the 4-minute timer, lift the rack out of the developer, tilt it toward one side, and allow the rack to drain into the developer tank.

When time is up, place the rack of developed films into a running water bath for 90 seconds. Agitate constantly.

With 5 seconds left on the 90-second timer, drain the negatives and place the rack into room temperature Working Fixer for twice the time it takes the negatives to "clear". That is, the milky, unreduced silver halide molecules will become dissolved into the alkaline Fixer solution over time. The fresher the Fixer, the faster the time it takes to "clear" the silver halide from the negatives; the older the Fixer, the longer the time it takes to clear them. On average, the films should be left in the Fixer for 5 minutes. Agitate, as described earlier.

While the films are in the Fixer, you should place the next rack of films in the running water wash, and begin the cycle again for that next rack.

When the first films have been thoroughly fixed, they must be washed for 30 minutes in running water. A single capful of Kodak Photoflo, diluted in a one-gallon container, should be prepared. Film racks should be dipped into the photoflo solution for 20 seconds prior to air drying. This helps prevent the formation of water spots. A convection oven set to 30 to 60 degrees C will speed the drying process. Left alone to dry by themselves, the negatives should be ready (dry enough) to be placed into protective envelopes after 60 to 120 minutes.

At times when water conservation is in order, the films can be placed into a working solution of Kodak Hypo Clearing Agent (or equivalent) for one minute after a one minute wash, then rinsed once more for one minute, and dried.

Alternatively, the racks of film can be placed in the fume extractor cabinet. The moving current of air will quickly dry them.

When all film has been developed and fixed, the room lights may be turned on. Before concluding your session, return to safelight conditions and reload the empty film frames with fresh, unexposed film. Refill the unexposed film canister and place the covered canister into the TEM's film 6esiccators. **Never, under any circumstances, place non-desiccated film into the TEM camera chamber**. The TEM will take hours to pump down and you risk contaminating the diffusion pump oils with water. Film is considered desiccated after 12 hours of pumping under a rough pump.

END SOP 305

SOP No. 304

TITLE: **SAED INDEXING PROCEDURE**

Guidelines for Using the Monoclinic and Orthorhombic Crystal Worksheets for the Indexing of Diffraction Patterns

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Approved & Authorized: _____

G. Edward Carney, Technical Manager

Approved & Authorized: _____

Andreas Saldivar, Laboratory Director

Approved & Authorized: _____

Joseph Coco, QA Manager

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 304 TITLE: **SAED INDEXING PROCEDURE**
Guidelines for Using the Monoclinic and Orthorhombic Crystal Worksheets for the Indexing of Diffraction Patterns

Sign: _____ Date: 6/23/10

Print Name: G E Carney

Sign: _____ Date: 6/24/10

Print Name: Andrew Soldivar

Sign: _____ Date: 6/24/10

Print Name: Amy Cao

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 304 SAED Indexing Procedure – Guidelines for Using Monoclinic and Orthohombic Crystal Worksheets for the Indexing of Diffraction Patterns

Sign: _____ Date: 10/30/12

Print Name: _____

Sign: _____ Date: 10/30/12

Print Name: Michael Greenberg

Sign: _____ Date: 10/30/12

Print Name: ROBERT PRIVETTE

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 304 SAED Indexing Procedure – Guidelines for Using Monoclinic and Orthohombic Crystal Worksheets for the Indexing of Diffraction Patterns

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Guidelines for Using the Monoclinic and Orthohombic Crystal Worksheets for the Indexing of Diffraction Patterns

INTRODUCTION

These instructions are to assist in the indexing of diffraction patterns. A light box, magnified loupe, calculator with trigonometry functions, diffraction image negative, and copies of the monoclinic (or orthorhombic) diffraction pattern worksheet are necessary to index patterns. These instructions are to be used as a guide and not to be considered as a substitute for formal, classroom training.

The masters of the monoclinic and orthorhombic forms are kept in the master form "pink book".

An alternate method and faster method of indexing amphibole diffraction patterns, using Dr. Su's tables, is described at the end of this section.

INSTRUCTIONS

Enter the negative number, camera length, analyst name, and date in the spaces provided.

To determine the camera constant, measure at least 4 equally spaced radii and enter in the spaces provided. For example, the first radius measurement, in millimeters, would be entered at the r1=_____mm. After 4 measured radii are entered, the average is computed and placed in the appropriate space. If a gold (Au) sample is used as a standard, multiply this number by 2.355 Angstroms to compute the camera constant. For other standards, use an appropriate d- spacing distance for that standard.

If using the form to index a diffraction pattern, write the known value of the camera constant in the space provided.

I. Place the center "cross hair" of a calibrated loupe over the center spot of the diffraction pattern. Orient it so that the ruler lays across the row of spots whose inter-spot distance is the smallest; a small distance in reciprocal space will equal a large distance in real space–an advantage!

WITHOUT MOVING THE LOUPE: Measure the distance from the center to the farthest spot to the "left" of the ruler's center; enter value in the space marked "D1 = _____mm".

WITHOUT MOVING THE LOUPE: Measure the distance from the center to the farthest spot to the "right" of the ruler's center; enter value in the space marked "mm + _____ /". (to the right of the previous space)

Count the number of "inter-planar distances" measured (equal to the number of spots measured, minus 1) and enter it in the "mm/ _____ =" space, provided.

Add the distances and divide, and enter the quotient in the " =_____mm " space. This is the average inter-planar distance of the unit cell (in real space).

Using a straight-edge ruler, draw a line through all of the spots along the plane observed. Extend the line in both directions, to the far edges of the negative envelope.

II. Repeat this procedure for another row of spots. This row must also cross the center spot and should ideally have the 2nd-smallest distance between them.

Optionally, repeat this for a third row (the "row" on the "diagonal", usually). This step is usually not performed unless confirming your results of the entire procedure (step VI).

III. Divide each of the "d" spacings calculated into the camera constant to yield a measured d-spacing for each row, in Angstroms.

Use these 2 measured d-spacings and refer to the pages of the Miller Index cards, containing indices for thousands of minerals. Fortunately, the search is confined to known asbestiform minerals and minerals commonly found with them, such as gypsum.

For example, if the EDXA information showed that the structure- in-question contained classic chemistry of Amosite, the known d-spacing values ("d-spacings", for short) for Amosite, would be examined. Look through the columns of numbers to find a match; if the measurement was 5.27A or 5.13A, the (001) crystal plane may be present. Parentheses indicate planes. The Miller index says that 5.20A is the distance that identifies the (001) plane. Allow for error (5% is usual).

Once a tentative identity of two crystal planes is made, the respective indices are entered in the spaces provided.

If you are using Dr. Su's tables to index you jump from this point to the alternate method after step VIII.

IV. Now, the indices are transferred to the "h1= ____ k1= _____ l1= _____" region of the worksheet. The "h1" represents the first number of the first 3-digit index (the '0' of the index (001)), the "k1" represents the second digit of the index (the middle '0' of our example) and the "l1" represents the third number of the index (the '1'). The potative index for the second vector is then transferred to the worksheet (use spaces provided for "h2, k2, and l2").

N.B. The "l" in the "l1" and l2" is a lower-case "L". Note also that for monoclinic crystals, the value of the sum of "h" and "k" must always be EVEN. Sums that are ODD will not be visible on the electron micrograph due to the phenomenon of destructive interference.

Returning to the Miller Index card, copy the unit cell parameters to the appropriate spaces on the worksheet: a , b , and c on the cards have numbers associated with them that are the interplanar distances for the 3 zone axes of that crystal. Amosite, for example, has values 9.356 for a, 18.380 for b, and 5.338 for c. (These values are in A)

Now, copy the value for the angle between the a and c planes. This is designated as B or (beta). For Amosite, it is 101.86 degrees. For monoclinic crystals, angles alpha and gamma are 90 degrees. BE SURE THAT THE VALUE OF THE ANGLE IS IN DEGREES, NOT DEGREES/HOURS/MINUTES, OR RADIANS. DURING SUBSEQUENT CALCULATIONS, BE SURE THAT THE CALCULATOR IS OPERATED IN THE "DEGREES" MODE AND NOT THE MODE FOR RADIANS.

V. Plug in the values for each of the formula's variables and solve for cosine of theta (cos). The formulas will compute the cosine of the angle between the 2 planes whose d-spacings were measured on the diffraction pattern. To compute the actual angle, obtain the arccosine of the cosine value.

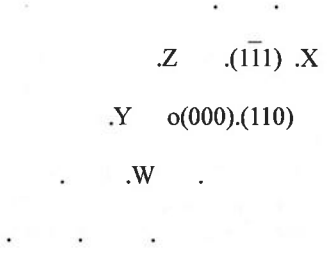
VI. Measure the angle between the 2 planes drawn on the pattern, and compare it to the computed angle. If there is a match (allow for possible measurement error), then confirm by measuring the average d-spacing of a third set of spots (the "Optional" D3 measurement and computation, referenced in section II), compute the Miller index of the closest spot of the third plane to the center spot [use vector addition for this] and compare the computed d-spacing that corresponds to that spot/index combination. If everything meets the minimum criteria, then proceed with the actual indexing of the pattern.

N.B. If the computed angle = (180 minus the measured angle) then check the sign of the relative Miller index value; if the tentative index is (001) try using $(00\bar{1})$ instead, then recalculate the angle.

Negative numbers are displayed with a bar over the number instead of a dash to the left of the number.

VII. Draw the diffraction pattern (or use a positive, photographic print) and write the Miller index for at least 10 of the closest spots to the center origin. This is called "indexing the pattern".

Indexing the pattern requires knowledge of vector addition.



"o" represents the center, or "origin" of the pattern

The index of the spot represented by the X would be the addition of the vectors to its left and below:

$(1\ \bar{1}\ 1) + (1\ 1\ 0)$

This is done by adding the values of each "like" element:

(1+1), (1+1), and (-1 + 0). This yields the index (221).

The index of the spot represented by the letter Y is the negative of the index (110), or $(\bar{1}\ \bar{1}\ 0)$.

In fact, all indices along the "perpendicular" lines drawn through the origin are complementary. Therefore, the index of the spot represented by the letter W is the negative of the complementary spot $(1\ 1\ \bar{1})$: (1 1 1). So, the index of the spot represented by the letter Z can be computed as $(1\ \bar{1}\ 0) + (1\ 1\ \bar{1})$, which equals $(0\ 0\ \bar{1})$.

VIII Compute the zone of the pattern. To accomplish this, use the vector multiplication rule:

Choose any two indices. Write the first index down, and write it down again, immediately to the right. For example, if the first index chosen happens to be (110), then write "1 1 0 1 1 0".

Write down the second index, "twice", directly beneath the first set of numbers. For example,

if the second index chosen is (111), then you should write "1 1 1 1 1 1" under the "1 1 0 1 1 0". The array should appear this way:

1 1 0 1 1 0

1 1 1 1 1 1

Place a vertical line to the right of the first column of numbers in the array, and a second vertical line to the left of the sixth column of numbers:

1 1 0 1 1 0

1 1 1 1 1 1

Cross multiply, using standard matrix-multiplication format: If the numbers in the array were letters as this diagram illustrates:

a b c d e f

g h i j k l

Then follow the formula: (b*i) – (c*h) to compute "u"

(c*j) – (d*i) to compute "v"

(d*k) – (e*j) to compute "w"

The zone of the pattern is defined as [u v w]. Brackets denote a zone. In this example,

(1*1) – (0*1) = 1-0 = 1

(0*1) – (1*1) = 0-1 = -1

(1*1) – (1*1) = 1-1 = 0

The zone = 1 -1 0 and is represented by the following notation:

$[\ 1\ \bar{1}\ 0\ ]$

Remember: negative numbers, whether representing planes in real space (h k l), reciprocal space (h* k* l*), vectors [u v w]*, or zones [u v w], are displayed with a bar over the number instead of a dash to the left of the number.

Alternate Method.

After measuring the d-spacings for two rows, as described through section III, measure the angle between the rows. This angle is referred to as "Theta".

Next refer to Dr. Shu-Chun Su's d-spacing and interfacial angle tables for amphibole asbestos minerals. These tables contain all the possible combinations were the theta angle is greater than 45 degrees.

Compare your largest d-spacing to the table and find the a match for your second d-spacing. If the theta angle in the table is within 5% of the angle you measured you have a match and the indexing is complete. Many of the d-spacings differ in length by small amounts. If your first try did not match be sure to completely check the tables.

Note: The values listed in the table are for zone axis patterns. If your photo is not a zone axis pattern the tables will not match. If your photo is close to a zone axis but not exact the tables will be slightly off. In this case you should still be able to match values.

If you know that your photo is a good zone axis pattern and you cannot index the pattern using either method, the mineral is most likely not asbestos even if the chemistry matches.

END SOP 304

SOP No. 303

TITLE: **HOW TO GET A DIFFRACTION PATTERN ON THE TEM**

Approved & Authorized: _____

G. Edward Carney, Technical Manager

Approved & Authorized: _____

Andreas Saldivar, Laboratory Director

Approved & Authorized: _____

Joseph Coco, QA Manager

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 303 TITLE: **HOW TO GET A DIFFRACTION PATTERN ON THE TEM**

Sign: _____ Date: 6/24/10

Print Name: _____

Sign: _____ Date: 6/23/10

Print Name: G E Carney

Sign: _____ Date: 6/24/10

Print Name: Andreas Saldivar

Sign: _____ Date: 6/24/10

Print Name: Ang Cao

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 303 How to get a Diffraction Pattern on the TEM

Sign: Michael Greenberg Date: 10/30/12

Print Name: Michael Greenberg

Sign: Robert Privette Date: 10/30/12

Print Name: ROBERT PRIVETTE

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 303 How to get a Diffraction Pattern on the TEM

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

To obtain an indexable diffraction patter on the JEOL100CXII the particle under consideration must be centered and at eucentric height. This is done at the beginning of the analysis and adjusted for each particle. To adjust the eucentric height:

1. center the particle,
2. focus on the particle,
3. tilt the stage in one direction,
4. if the particle moves away from center, then bring it back with the height adjusting knob,
5. tilt the stage back to zero degree tilt,
6. focus on the particle,
7. tilt the stage in the same direction of step 3,
8. center the particle, if it moves, again bring it back with the height adjuster,
9. tilt the stage back to zero degree tilt,
10. focus on the particle, and
11. repeat steps 3 to 10 until the particle remains relatively centered when tilting.

The illumination is decreased as much as possible by rotating the C_2 knob in a clockwise direction. The particle is focused during either the Fresnel fringe or the image wobbler. The objective aperture is removed. The selected area diffraction mode (SAED) aperture is inserted. Switch the TEM to selected area lens mode. Focus field limiting aperture with small knob of SA magnification control. Focus particle with focus knob. Switch the TEM from the SA imaging mode to the diffraction mode. To focus the diffraction pattern, adjust the diffraction focus knob until the central beam is as small as possible and symmetrical. Alternatively one may insert the objective aperture and focus on it, then remove the aperture. Introduce the beam stop to the diffraction pattern and center it on the transmitted beam, which is in the center of the pattern. If the pattern is very faint you can adjust the illumination to obtain brighter spots. This should be done sparingly because: the image may shift due to charging, thus eliminating the diffraction pattern and if photographing is desired (described below) then decreasing the illumination again is required which may cause shifting again.

The camera length may be adjusted to decrease or enlarge the diffraction pattern. In routine asbestos analysis you will find that there are generally two settings used most often. One setting is for the small screen and binoculars and the other setting is for photographing, which is determined from reference materials. Usually the pattern requires refocusing when changing the camera length. Also the center of the pattern may shift. This is the reason that the "two settings" mentioned earlier were presented because usually at least two camera length settings can be aligned so that their centers very closely coincide.

Once an SAED pattern has been obtained, the desired camera length chosen and focused, and the pattern centered, then it may be photographed. Photographing electron diffraction patterns is an art. It is done in the manual mode, i.e., the exposure time cannot be automated in most scopes. For the first SAED micrographs it is best to start with a standard. Obtain a pattern as described above. Decrease the illumination completely, i.e., turn the C_2 lens knob fully clockwise. The diffraction pattern will not be visible at this point. This technique allows for a parallel beam of electrons eliminating any convergence and produces very crisp spots. Take photographs starting at 0.5 second exposure, doubling the exposure time for each micrograph until 2 or 3 minutes are attained. Develop the negatives according to normal darkroom procedures. If there is not a usable negative then it is likely that a longer exposure time is needed. Repeat the process described above until a satisfactory negative is produced. Once an acceptable exposure time has been found, keep a mental note of it.

After the negatives are developed then the diffraction pattern can be measured and indexed (see Sections 6 and 11). For measuring it is ideal to "burn" a spot on top of the beam stop. This is done so that the center spot can be used in the measurements. Double exposure techniques are used to burn the central spot. The first exposure is of the SAED pattern with the beam stop in place. The second exposure is taken after the beam stop is removed and usually is exposed for 0.2 to 0.5 seconds. Sometimes a triple exposure is needed, for example in calibrating the lens rotation angle between calibration and image modes. See Section 11.

Once a diffraction pattern is obtained the row spacing can be directly measures on the screen by using a calibrated objective aperture.

The objective aperture is calibrated by measuring it's diameter on the camera constant photograph. When photographing the gold rings for the camera constant a multiple exposure is used. The first exposure is of the rings for 45 seconds. This is with the central stop covering the central diffraction spot. The second is exposure is for .12 seconds with the central stop removed.

To photograph the objective aperture another exposure of 45 seconds is used. When the first exposure of the gold rings is shot, objective aperture 2 or 3 is inserted and moved away from the center of the photo. After the exposure is down the objective aperture is removed and the other 2 exposures are taken. This results in a negative with a circular area of longer exposure representing the diameter of the objective aperture at that camera length. You can then calculate how many 4.3 angstrom rows would be visible at that camera length with the objective aperture inserted. It is usually 4.6 to 4.7 rows.

END SOP 303

SOP No. 302

TITLE: **GRID OPENING ORIENTATION, SELECTION AND TRAVERSE**

Approved & Authorized: _____

G. Edward Carney, Technical Manager

Approved & Authorized: _____

Andreas Saldivar, Laboratory Director

Approved & Authorized: _____

Joseph Coco, QA Manager

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 302 TITLE: **GRID OPENING ORIENTATION, SELECTION AND TRAVERSE**

Sign: _____ Date: 6/22/10

Print Name: Mark Craighead

Sign: _____ Date: 6/23/10

Print Name: G E Carey

Sign: _____ Date: 6/24/10

Print Name: Andrea Saldivar

Sign: _____ Date: 6/24/10

Print Name: Amy Cano

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 302 Grid Opening Orientation, Selection and Traverse

Sign: Michael Greenberg Date: 10/30/12

Print Name: Michael Greenberg

Sign: Robert Privette Date: 10/30/12

Print Name: ROBERT PRIVETTE

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 302 Grid Opening Orientation, Selection and Traverse

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

GRID ORIENTATION

Grids are placed carbon side up in single tilt sample holders and carbon side down in Gatan double tilt holders.

The grids are oriented so that the indexing grid pattern is substantially parallel to the direction of traverse. Allowance is made for in-exact parallel placement by the operator through a careful traverse so that all areas of the grid are analyzed (see **GRID TRAVERSE**).

Grid placement for verified counting follows the protocol outlined in the NVLAP proficiency instructions pages 11-13, dated January 25, 1990. Essentially, the letter "F" is viewed at the working magnification in the upright orientation. Correct grid placement is checked by scanning a grid opening from the upper left to the lower left corners. When correctly placed, the left-hand edge of the grid bar remains in the field of view for an entire traverse.

GRID OPENING SELECTION

Evaluation of the overall grid preparation is specified in the analytical section of this manual. When grid preparations meet the evaluation criteria, the grid openings selected for analysis are randomly chosen.

It is important that openings are chosen from different grid quadrants and that adjacent grid openings are not consecutively analyzed.

GRID TRAVERSE

When analyzing a grid opening, the traverse begins in the upper left hand corner and proceeds downward. When a single traverse is completed the horizontal stage control is used to move the grid one field of view. The next traverse is completed in the opposite direction (upward); pattern continues until the entire grid opening area has been analyzed.

When the grid placement is not parallel to the direction of traverse, the operator ensures that the entire area is analyzed.

Grid indexing designations are shown on the attached sheet.

END SOP 302

SOP No. 301

ALIGNMENT PROCEDURE – JEOL 100CX ELECTRON MICROSCOPE

Approved & Authorized: _____

G. Edward Carney, Technical Manager

Approved & Authorized: _____

Andreas Saldivar, Laboratory Director

Approved & Authorized: _____

Joseph Coco, QA Manager

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 301 ALIGNMENT PROCEDURE – JEOL 100CX ELECTRON MICROSCOPE

Sign: _____ Date: 6/22/10

Print Name: _____

Sign: _____ Date: 6/23/10

Print Name: GE Carney

Sign: _____ Date: 6/24/10

Print Name: Andreas Saldivar

Sign: _____ Date: 6/24/10

Print Name: Ang Cao

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 301 Alignment Procedure – JEOL100CX Electron Microscope

Sign: Michael Greenberg Date: 10/30/12

Print Name: Michael Greenberg

Sign: Robert Privette Date: 10/30/12

Print Name: ROBERT PRIVETTE

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 301 Alignment Procedure – JEOL100CX Electron Microscope

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

With all but the Condenser aperture withdrawn and no specimen, turn H.V. on to 100kV, generate a beam, and find the beam with gun tilt and translate controls.

1. **FILAMENT ALIGNMENT** - With beam on screen set Mag. at 4,800x, condenser aperture at 2 or 3, Spot Size at 1, condenser at cross-over, center the spot with gun translate controls. Desaturate filament and center filament image in halo with gun tilt controls. Resaturate filament. Beam current at cross-over should be 25 microamps above the dark current level for optimum use.

2. **GUN/CONDENSER ALIGNMENT** - Switch to Spot Size 3. Bring Condenser to cross-over and center beam with beam align. trans. knobs. Switch to Spot Size 1 again, find cross-over and recenter beam with gun translate controls. Repeat until spot centers coincide. Go to spot size 1 and recenter gun tilt as in Step 1.

3. **CONDENSER APERTURE CENTERING** - Insert desired condenser aperture, find cross-over and center the spot on the screen with beam align. trans. knobs. Spread the beam and center circle of illumination on the screen using aperture X and Y translation controls.

4. **CONDENSER LENS STIGMATION** - With Spot Size 3, condenser at cross-over, and Mag. at 19,000x, adjust condenser stigmator controls to obtain a symmetrical spot. This may also be accomplished by desaturating the filament and adjusting the condenser stigmator controls to obtain a sharp image of the striations in the center of the filament image. Switch back to spot size 1, center beam, and continue with alignment.

   a.* **CONDENSER STIGMATOR BALANCING** - If the beam moves while stigmating, turn 'X' stigmator to one side of center and note movement of the beam. Return beam to center with COND STIGMATOR X1, X2 controls on lower right panel. Repeat for 'Y'.

5. **Z AXIS CORRECTION** - Insert a specimen. Locate and center an object at a Mag of 19,000x and focus. Tilt the stage (CCW) and note movement of the object. Return it to the center with the Z adjust knob. Return the stage to horizontal, refocus and repeat, tilting further each time until object remains centered with minimal sweep.

   a.* **STAGE CENTERING** - Tilt the stage in the opposite direction and note movement of the object. Return object to center using the three (3) stage adjust hex screws on the column. Tilt back to horizontal, focus, center object with stage drives and repeat steps 5. and 5a. until the object remains centered through full tilt on both sides of horizontal.

6. **DEFLECTOR COIL BALANCING** - Reduce Mag. to 5,000x or less, focus image and bring condenser to cross-over. On lower left panel, switch Wobbler up to 'X' and converge the 2 illumination spots on the screen with 'X' Compensator and Corrector knobs. Repeat for 'Y'. Increase Mag. to 19,000x and repeat.

7. **IMAGE WOBBLER BALANCING** - With the specimen focused at 19,000x, bring the beam to crossover and turn on the IMAGE WOBBLER switch. Converge the two beams with the IMAGE WOBBLER 'A' and 'B' knobs on lower right panel.

8. **OBJECTIVE LENS ALIGNMENT** - Align the objective lens by one of the following procedures:

   a. **VOLTAGE AXIS CENTERING** - For magnification of 19,000x or greater - Locate a small point object in the specimen and center it on the screen. Spread the beam to a comfortable level. Switch on the H.V. Wobbler and note direction of image movement. Minimize

movement by adjusting beam align. tilt knobs to move deflected image back to center. Recenter illumination with beam align. trans knobs as necessary.

b. **CURRENT AXIS CENTERING** - for magnification below 30,000x - Locate a small point object in the specimen and center it on the screen. Under focus the image four counterclockwise steps on the outer Medium Focus knob and note the movement of the image. Re-center the image with the beam align. tilt knobs, refocus, and repeat until the image does not shift during focusing. Recenter illumination if necessary with align. trans. knobs.

9a.* **BEAM COMPENSATION / FOCUS** - Using Spot Size 2, condenser at cross-over, focus an image at 1 0,000x - 30,000x. Using the lower Medium focus control, over focus the image and center illumination with BEAM COMPENSATOR SCREWS. Under focus image and center illumination with beam align. trans. knobs. Repeat until beam remains centered.

9b.* **INTERMEDIATE / PROJECTOR LENS AXIS ALIGNMENT** - With the Magnification at approximately 19,000x and specimen focused, insert the diffraction aperture used most and center it. Depress SA Mag button and focus aperture. Depress SA Diff. button, set Camera Length to position 22 cm (full CCW), and focus the spot. Center projection spot on screen with upper set of Hex screws. Switch Camera lens to 120 cm from full CCW and center spot with lower set of Hex screws. Repeat until spot centers coincide. Return to Mag. conditions by inserting and centering objective aperture and removing diffraction aperture.

9c.* **ALTERNATE METHOD** - At 19,000x, go the crossover and center the beam. Switch to SA Diffraction mode, at 330 cm (1 click stop before full clockwise position). Use diffraction focus knob to focus the caustic image to a spot (or, if a crystal is under the beam, focus the diffraction pattern). Center the spot using the intermediate lens alignment Hex Screws. Switch camera length to 55 cm (1 click stop before full counterclockwise position). Use diffraction focus knob to focus the caustic or diffraction spot. Repeat alignment between 330 and 55 cm until minimum movement is seen between the two. Return to Mag mode, and view position of crossover spot: it should still be in the center of the screen. Return to SA DIFF modeand select the camera length you use most for photography or video recording. Center focused spot with the projector lens adjustment screws. Continue with Scope Alignment.

10. **OBJECTIVE APERTURE CENTERING** - Insert Diffraction aperture #1 and switch function to SA diffraction focus diffraction spot. Insert desired objective aperture. Center aperture with respect to beam spot using aperture X and Y controls. Remove diffraction aperture and switch Function to Mag. At sufficiently low mags, the aperture may be centered directly from its image on the screen.

11. **OBJECTIVE LENS STIGMATION** - select stigmator control set #1 or #2 by the switch on the lower left panel. Stigmate, using a holey carbon substrate, with coarse and fine X and Y controls on left panel. Use a mag at least as high as the maximum to be used routinely, but preferably over 100,000x , and spread the beam until the image is barely visible through the binoculars.

12.* **OBJECTIVE STIGMATOR BALANCING** - If the image moves while stigmating, turn 'X' stigmator to one side of center and note movement of the image. Return image to center with OBJ STIGMATOR X1, X2 controls on lower left panel. Repeat for 'Y'.

END SOP 301

Approved & Authorized: _____
Andreas Saldivar, Laboratory Director

Approved & Authorized: _____
Joseph A. Coco, QA Manager

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 206 Laboratory Blank Contamination Procedures

Sign: _____ Date: 2/01/13

Print Name: Ed Carney

Sign: _____ Date: 1/31/13

Print Name: Ang Cao

Sign: _____ Date: 1/31/13

Print Name: Michael Creaghan

Sign: _____ Date: 1/31/13

Print Name: Michael Greenberg

Sign: _____ Date: 2/1/31

Print Name: Robert Privette

Sign: _____ Date: _____

Print Name: _____

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 206 Laboratory Blank Contamination Procedures

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

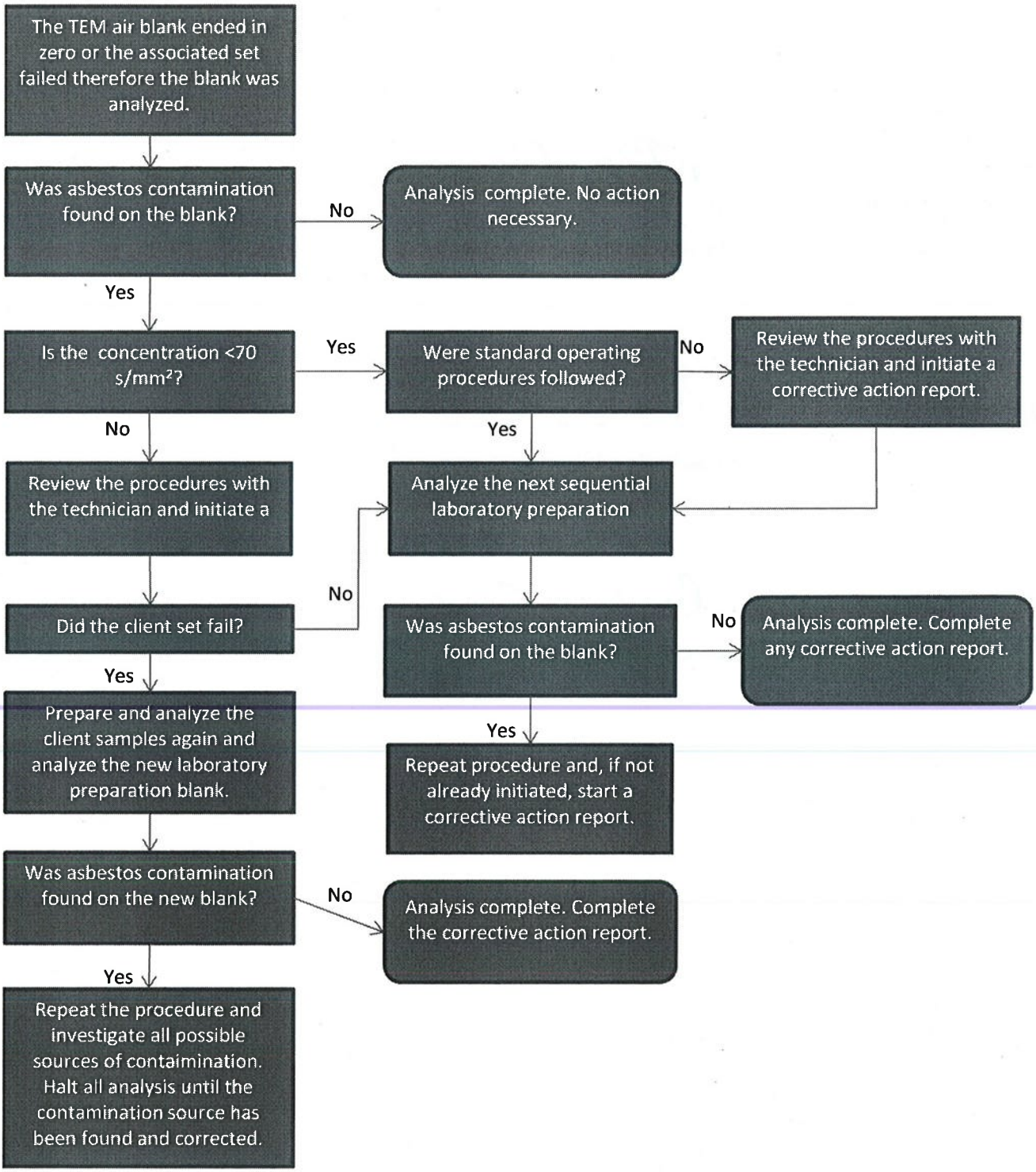
Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____


The TEM air blank ended in zero or the associated set failed therefore the blank was analyzed.
Was asbestos contamination found on the blank?
No
Analysis complete. No action necessary.
Yes
Is the concentration <70 s/mm²?
Yes
Were standard operating procedures followed?
No
Review the procedures with the technician and initiate a corrective action report.
No
Yes
Review the procedures with the technician and initiate a
Analyze the next sequential laboratory preparation
No
Did the client set fail?
Was asbestos contamination found on the blank?
No
Analysis complete. Complete any corrective action report.
Yes
Yes
Prepare and analyze the client samples again and analyze the new laboratory preparation blank.
Repeat procedure and, if not already initiated, start a corrective action report.
Was asbestos contamination found on the new blank?
No
Analysis complete. Complete the corrective action report.
Yes
Repeat the procedure and investigate all possible sources of contaimination. Halt all analysis until the contamination source has been found and corrected.

SOP No. 205

TITLE: CLEANING STAINLESS STEEL GRIDS

Approved & Authorized: _____

G. Edward Carney, Technical Manager

Approved & Authorized: _____

Andreas Saldivar, Laboratory Director

Approved & Authorized: _____

Joseph Coco, QA Manager

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 205 TITLE: CLEANING STAINLESS STEEL ~~GRIDS~~ Screens

Sign: _____ Date: 6/22/10

Print Name: Michael Geraghty

Sign: _____ Date: 6-22-10

Print Name: LAMAI WILLIAMS

Sign: _____ Date: 6-22-10

Print Name: CHON SIMPHA

Sign: _____ Date: 6/23/10

Print Name: GE Carney

Sign: _____ Date: 6/24/10

Print Name: Andreas Saldivar

Sign: _____ Date: 6/24/10

Print Name: Ang Co

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 205 Cleaning Stainless Steel Grids

Sign: _____ Date: Oct 30, 2012

Print Name: ROBERT PRIVETTE
Bob

Sign: _____ Date: 10/30/12

Print Name: KRISHA ORDANIEL
Krishe

Sign: _____ Date: 10/30/12

Print Name: Michael Greenberg
Mike G

Sign: _____ Date: 11/8/12

Print Name: Barbie Vanlerberg

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

During final filter dissolution, grids and filter sections are laid directly onto the stainless steel mesh of the modified Jaffe wick washer. Therefore, care must be taken to ensure that these screens are thoroughly cleaned following each use.

The cleaning procedure involves several steps. First, the screens are placed into hot water with liquid detergent, scrubbed with a nylon bristle brush, and rinsed thoroughly to remove any detergent remnants. Fiber-free water is used as a final rinse. The screens are then placed into a disposable plastic beaker, covered with acetone, and placed in an ultrasonic cleaner for five minutes. Following this, the screens are rinsed with fresh acetone and allowed to air dry in a fume hood.

END SOP 205

Zepto Plasma Asher

1. Open the main valve on the O_2 tank. If necessary, adjust the regulator to achieve a pressure of 5-10 PSI.
2. Turn on (VCT) power converter.
3. Turn main power on.
4. Put the slides the chamber.
5. Place the cover over the chamber and hold it in place.
6. Turn on the power switch on the left side of the cart. This turns on the vacuum pump.
7. Release the cover when you feel the vacuum is holding it in place.
8. Wait for the vacuum gauge to read 0.3 mbar or less.
9. Adjust the Gas2 dial until reads between 50 and 60. Note: this gauge is sensitive.
10. If necessary, adjust the power dial to 65% and the timer to 1 minute.
11. Turn the generator on. Note: a blue plasma indicates oxygen. A purple plasma indicates air. The desired plasma is blue.
12. When the generator stops, turn the Gas 2 dial until reads zero.
13. Turn off the switch on the cart. This turns off the mechanical pump.
14. Hold the chamber cover and push the ventilation button. When the cover releases, place it on top of the asher.
15. Turn off the ventilation button.
16. Turn off the main power and the power converter.
17. Close the valve on the O_2 tank.

~~2.~~

AAA Arts Plasma Asher

* Turn vacuum pump power on.
* Switch Asher AC **ON**, turn vacuum switch **ON**. Allow unit to warm up for 5 minutes before use.
* Open oxygen tank main valve (5 psig) at least 5 minutes prior to inserting first sample.
* Switch vacuum off, wait for chamber seal to release (approximately 2-3 minutes).
* Carefully remove sample chamber.

[A] * Insert one slide preparation into chamber, placing it evenly over second and third venting slit from the solid face of the chamber, making sure venting slits are in the down (4 o'clock to 8 o'clock) position. Note: 1 slide preparation may contain many filter segments. If additional slide is to be ashed at the same time, place it directly behind the first slide.

* Insert asher into chamber housing and switch vacuum to **ON**. Make sure chamber is centered in housing, creating an excellent vacuum seal.

* Evacuate chamber 2 minutes while oxygen is flowing into chamber.

* Set timer for 90 seconds (or new or calibrated value). Turn on RF power and meter switches.

* Adjust RF power level to setting 4. Adjust tuning (reflected power) to obtain lowest power level. Readjust RF power level to full position.

(OPTIONAL): * Turn out lab lights in order to view the burning plasma, and switch RF power **ON**.

* Adjust tuning until a bright blue plasma is detected.
NOTE: If a violet plasma is obtained, there is nitrogen in the chamber. Pump chamber for at least five minutes prior to ashing.

* Start timer. The amount of time needed to etch 10% of the filter is determined by trial, at each laboratory.

* Adjust tuning until blue plasma is at its brightest, and leave it there.

* After the designated time has elapsed, switch RF power and meter **OFF**.

* Switch vacuum **OFF**, wait for chamber seal to release, then remove sample chamber.

* Carefully remove sample, avoiding drafts or any disturbance that may blow off particulate material, and transfer the slide in covered petri dish to the carbon coater.

* To repeat the procedure for additional slides of samples, go to set [A].

* Replace sample chamber, switch vacuum to **ON**. Turn oxygen supply **OFF**.

* When chamber has pumped down for approximately one minute switch AC **OFF**, and turn off vacuum pump.

CALIBRATION PROCEDURE

The calibration procedure is performed annually or after RF tube changing, by the gravimetric method.

Representative filter sections are affixed to a pre-weighed glass microslide.

The slide is reweighed to obtain the weight of the filter(s).

The slide is placed in the asher and the unit is evacuated for a period of ten minutes. The slides are then reweighed to determine if additional weight loss caused by the evaporation of the collapsing solution has

occurred. When this weight loss becomes negligible (usually 10 minutes), the slide is etched to determine 10% weight loss of filter.

The slide is repeatedly etched, equilibrated (brought to STP and humidity) then weighed. When the weight calculations yield results indicative of a 10% weight loss of the filter, the total etching times are summed and used as the standard etching time.

Often it is found that ashing a filter until 10% of the filter ~~is removed~~is removed produces an undesirable result. The filter, when prepared and viewed under the TEM appears pock marked and uneven. This is a result of over ashing. An alternative method of plasma asher calibration involves preparing a number of slides and ashing each for a different amount of time, 1 minute, 2 minutes… The filters from each slide are prepared and viewed under the TEM. The sample that has the best appearance, even orange peel texture, determines the amount of time used for ashing.

END SOP 204

Approved & Authorized: _____

G. Edward Carney, Technical Manager

Approved & Authorized: _____

Andreas Saldivar, Laboratory Director

Approved & Authorized: _____

Joseph Coco, QA Manager

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 203 Directions for Carbon Coating (with optional Gold Evaporation Procedure)

Sign: _____ Date: 6/22/10

Print Name: Michael Creighton

Sign: _____ Date: 6-22-10

Print Name: Chon Simpher

Sign: _____ Date: 6/23/10

Print Name: G E Carney

Sign: _____ Date: 6/24/10

Print Name: Andreas Saldiver

Sign: _____ Date: 6/24/10

Print Name: Amy Cano

Sign: _____ Date: 11/8/12

Print Name: Bonnie Vanlent

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 203 Directions for Carbon Coating (with optional Gold Evaporation Procedure)

Sign: [signature] Date: 10/30/12

Print Name: ROBERT PRIVETTE
Bob

Sign: [signature] Date: 10/30/12

Print Name: KRISHA BRANNER
Krisha

Sign: [signature] Date: 10/30/12

Print Name: Michael Greenberg

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

For the Denton DV 502A.

From a Cold Start:

* Our Operators judge the thickness of the carbon film. If the film is too thin, large particles will be lost from the TEM specimen, and there will be few complete and undamaged grid openings on the specimen. If the coating is too thick, the filter will tend to curl when exposed to chloroform vapor and the carbon film may not adhere to the support mesh. Too thick a carbon film also leads to a TEM image lacking in contrast, and the ability to obtain SAED patterns is compromised. The carbon film is made as thinly as possible to remain intact over most of the grid openings. A "guide slide", coated with the correct amount of carbon, is prominently posted near each carbon coater to serve as a visual cue for proper carbon thickness.

* Be sure Backing Valve (BV), Roughing Valve (RV), and Main Valve (MV) are closed.

* Turn on water supply.

* Turn on cooling (maintain a flow rate of 1 liter/minute. Temperature should be skin temperature).

* Main Power **ON**.

* Mechanical Pump **ON**.

* Thermo-Couple switch to Foreline position.

* Turn Diffusion Pump (DP) **ON**.

* Fill Nitrogen Trap until it overflows (which will occur by filling funnel to rim, letting it drain, then filling it again). Refill after 20 minutes, then every 90 minutes until you turn off the DP.

* Start a 10' timer. 10' from now the DP will be ready to create a high vacuum; you should be at step [A] by then.

* Open chamber vent valve. Air may be filtered through a 25mm, .45 micron MCE filter. Currently vents at 10 lpm or less.

* Lift bell jar and turn it to lock it in an open position.

* Close chamber vent valve.

* Loosen electrode locks (**CAUTION** - these may be very hot if coating procedure has recently been done).

* Remove carbon rods (**CAUTION** - these may be hot also!).

* Sand the end of the flat-tipped carbon rod until it is smooth. (Use 200 grit sandpaper for smoothing).

* Sharpen the tip of a carbon rod in a carbon rod sharpener approximately 5-7mm in length, then sand tip to a beveled point.

* Replace carbon rods into holder.

* Tighten electrode locks after allowing for spring tension.
* Place specimen slides on doublestick tape, (affix to the turn table).
* Refer to gold coating steps 1 & 2 if gold evaporation is desired.
* Close bell jar.
* Regrease rubber bell jar gasket as needed.

[A] Carbon Coat:

* Be sure the diffusion pump (DP) has been **ON** for at least 10 minutes.
* Open Roughing Valve (RV).
* Wait until foreline reads less than 50 mTorr.
* Close RV.
* Open Backing Valve (BV). Wait for foreline gauge to fall near 50 mTorr or less.
* Set thermocouple switch to Chamber.
* Slowly open Main Valve (MV). The pressure on thermocouple gauge should fall near zero.
* Turn High Vacuum Range Select from the **ON** position to the 10^{-4} position, (you may have to press the METER READ button). A 1-minute wait may be required for warm up. Click 1 step at a time waiting until gauge reads below 1 for that scale.
* Set Filament Adjust knob at 0% filament power (fully CCW).
* Turn **ON** rotary power and adjust speed (10-20 rpm is satisfactory).
* Set the Fil/Glow select to Fil.
* Set the Filament Selection switch to Fil 1.
* Turn **ON** filament power.
* When vacuum reads at or below $1x10^{-5}$ mTorr, SLOWLY turn filament power up until meter reads approximately 25 amps. There should be a FAINT orange glow of the carbon rod(s). It is safe to observe this faint-type of glowing carbon.
* Protective eyewear must be used, face shield or goggles, to protect against possible implosion. The welders glass must also be used to protect against the intense light emitted by the carbon rods
* Increase power slowly to approximately 45 amps or when a faint sparking occurs.

NEVER LOOK DIRECTLY INTO GLOWING CARBON WITHOUT PROPER EYE PROTECTION !

* If no sparking occurs after 15 seconds, increase slowly by 1-2 amp increments. If too much occurs, decrease slowly until uniform, light, sparking is maintained.

* When coating PC filters, spark for 5 seconds, remove power to filament, let rods cool for 30 seconds, and repeat. This will prevent excess heat from cross-linking the plastic polymers.

* Adjust amperage to maintain light sparking until desired amount is applied to sample, or filament burns out.

* Refer to gold coating step #3 if gold evaporating is desired.

To Remove Specimen or Recoat:

* Adjust filament (Power) to **0**.

* Turn off filament power.

* Turn off rotary stage power.

* Turn off range select vacuum gauge (knob turned fully CCW).

* Close MV.

* Open chamber vent. Adjust flow to 10 lpm.

* Close BV.

* Lift bell jar.

* Close chamber vent.

* Remove specimens (or cover them while setting up to reevaporate).

* Gold evaporation for internal camera constant standard may be completed next.

Gold Evaporation

Gold wire may be evaporated onto the filters to serve as an internal camera constant, after the deposition of carbon.

When preparing carbon coater for a set of samples, use these additional steps for gold coating.

- Measure 4mm of gold wire. Bend into a loop and attach it to a tungsten basket.

- Mount basket to set of electrodes designed for thin filaments establishing a tight connection.

- When carbon coating steps have been completed, and before breaking vacuum of the bell jar, do the following:

  * Be sure the carbon electrodes fil adjust level is at zero.

* Switch fil selector to the appropriate filament position.
* Turn on fil power.
* Turn fil adjust level until Tungsten filament glows faintly (a dull, reddish orange). Adjust power level slowly, but smoothly until 15 microamps are achieved.
* Glow here for 15 seconds.
* Turn fil adjust to zero.
* Turn off fil power.
* Switch fil selector back to the position for carbon coating if desired.
* Close main valve, then vent chamber to remove specimens.

TITLE: DIRECTIONS FOR CARBON COATING WITH THE EMS 950

1. Turn the unit on. (The **power switch** is on the back right side of the unit.)

2. Lift the top of chamber. Inspect the interior of the chamber for carbon build-up. If there is a lot of build-up vacuum the chamber.

3. Loosen carbon rod locks.

4. Remove existing carbon rods.

5. Sand the end of the flat-tipped carbon rod with the sandpaper board until it is smooth. (If the rod is to short, throw it away and get a new one.)

6. Sharpen the tip of the other carbon rod in the carbon rod sharpener approximately 7.0 mm in length, then sand down the tip to a beveled point using the sandpaper board. You can check the size of the rod by inserting it in manual carbon rod sharpener that was provided with the system. You insert the rod into the sharpener and the tip of the carbon rod should be flushed with back end of the sharpener. (If the rod is to short to be used throw it away and get a new one.)

7. Place rods into carbon coater rod holders.

8. Place the rod with the flat end into the carbon holder with the spring and expose 10mm of the rod towards the center. Tighten just enough to hold the carbon rod in place. (Over tightening will cause the rod to break.)

9. Place the carbon rod with the beveled tip into the stationary carbon rod holder and depress the other carbon rod holder until the spring is pressed in all the way. Lock the carbon rod. Tighten just enough to hold the carbon rod in place. (Over tightening will cause the rod to break.)

10. Remove carbon dust from carbon rod holders using an aerosol duster.

11. Place the slides on to the rotating stage.

12. Lower the top of the chamber slowly. (Make sure the top sits down flat on the carbon coater.)

13. Press the "start" button on the front panel of the carbon coater to start the automated carbon coating process. As soon as you press the "start" button on the carbon coater, turn on the mechanical pump by pressing the on button on the surge protector that the mechanical pump is plugged in to. It will take the coater five minutes before it is ready for the next step.

14. Press the "down" arrow to start the automated carbon coating process.

15. After the carbon coater stop coating press the "stop" button on the front panel of the carbon coater and then press the surge protector off button. It will take the carbon coater about one minute to reach atmosphere.

16. Lift top slowly and take slides out.

17. Turn off the carbon coater if this is the last set of samples to be prepared today

END OF SOP 203

Approved & Authorized: _____

G. Edward Carney, Technical Manager

Approved & Authorized: _____

Andreas Saldivar, Laboratory Director

Approved & Authorized: _____

Joseph Coco, QA Manager

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 202 PC FILTER PREPARATION

Sign: _____ Date: 6/23/10

Print Name: GECarney

Sign: _____ Date: 6/24/10

Print Name: Andreas Saldivar

Sign: _____ Date: 6/24/10

Print Name: Anap Ce.

Sign: _____ Date: 11/6/12

Print Name: CHON SIMPHA
Chon

Sign: _____ Date: 6/2/12

Print Name: Michael Gaughan
Michael C

Sign: _____ Date: _____

Print Name: Barbara Vaudert
Krisha JNH Ben

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 202 PC Filter Preparation

Sign: _____ Date: Oct 30, 2012

Print Name: ROBERT PRIVETTE
Bob

Sign: _____ Date: 10/30/12

Print Name: KRISHA BROWN
Krisha

Sign: _____ Date: 10/30/12

Print Name: Michael Greenberg
Michael G

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

The exterior of each cassette is wet-wiped with fiber-free water to minimize contamination possibilities before the samples are taken into the class 100 clean hood.

SELECTION OF FILTER AREA FOR CARBON COATING:

Before preparation of the filters, a 75 mm x 50 mm microscope slide is washed and dried. This slide is used to support strips of polycarbonate filter (PC) during the carbon sublimation. Two parallel strips of double-sided adhesive tape are applied along the lengths of the slide. PC filters are easily stretched during handling, and cutting of areas for preparation is performed with great care.

The filter and the mixed cellulose ester (MCE) backing filter are removed together from the cassette and placed on a cleaned glass microscope slide. A portion of the filter from the center-most part of the filter is cut with a curved scalpel blade by rocking the blade from the point placed in contact with the filter. This process is repeated to cut a strip approximately 3 mm wide across the diameter of the filter. The strip of PC filter is separated from the corresponding strip of backing filter and carefully placed so that it bridges the gap between the adhesive tape strips on the microscope slide.

The filter strip is held with fine-point forceps and supported underneath by the scalpel blade during placement on the microscope slide. The Operator places several such strips on the same microscope slide, taking care to rinse and wet-wipe the scalpel blade and forceps before handling a new sample. The filter strips are identified by writing on the glass slide using a marker insoluble in water. After the filter strip has been cut from each filter, the residual parts of the filter are returned to the cassette and held in position by reassembly of the cassette.

Carbon coating of filter strips is in accordance with SOP No. 203 for Carbon Coating.

PREPARATION OF THE JAFFE WASHER FOR PC FILTER DISSOLUTION:

A clean washer consisting of a simple stainless steel bridge is used by this lab. Three layers of lens tissue approximately 1.0 cm x 0.5 cm are placed on the stainless steel bridge, and the washer is filled with chloroform to a level where the meniscus contacts the underside of the mesh, which results in saturation of the lens tissue. The saturated tissue will wick, through diffusion, solvent to the filter, and the dissolved filter molecules will, in turn, wick dissolved filter molecules through the tissue into the reservoir of solvent beneath the bridge.

PLACING OF SPECIMENS IN THE JAFFE WASHER:

The TEM grids are first placed on the square pieces of lens tissue on the bridge so that grids can be picked up, in groups of four or six with forceps. Using a curved scalpel blade, the Operator excises three 3 mm square pieces of the carbon-coated polycarbonate filter from filter strip. The squares are selected from the center of the strip and from two points between the outer periphery of the active surface and the center. A single piece of filter is placed carbon side up on an indexed TEM specimen grid. The grid assembly is placed onto the saturated lens tissue within the Jaffe washer. When carbon-coated grids are used, the filter is placed carbon-coated side down. Any number of separate pieces of lens tissue may be placed in the same Jaffe washer. The lid is then placed on the Jaffe washer, and the system is allowed to stand for several hours, preferably overnight. The Jaffe washer is thoroughly cleaned after every use.

CONDENSATION WASHING:

We have found that many polycarbonate filters will not dissolve completely in the Jaffe washer, even after being exposed to chloroform for as long as 3 days. This problem becomes more serious if filter polymers were cross-linked by overheating during the carbon coating process. The presence of undissolved filter medium on the TEM preparation leads to partial or complete obscuring of areas of the sample, and fibers that may be present in these areas of the specimen will be overlooked; this will lead to a lower than true asbestos concentration. Undissolved filter medium also compromises our ability to obtain SAED patterns.

Before grids are analyzed, they are carefully examined to determine whether they are adequately cleared of residual filter medium. It has been found that condensation washing of the grids for 60 minutes after an initial hour of Jaffe washer treatment with chloroform as the solvent, clears residual filter media. In practice, the piece of lens tissue supporting the specimen grids is transferred to the cold finger of a charged, heated condensation washer, and the washer is operated according to the manufacturer's directions for about 1 hour. If the specimens are cleared satisfactorily by the Jaffe washer alone, the condensation washing step is not performed. The Jaffe washer and condensate washer are thoroughly cleaned after each use with a cleaning solvent and filter deionized water.

The grids are air-dried and stored in labeled grid storage boxes.

END SOP 202

Approved & Authorized:

G. Edward Carney, Technical Manager

Approved & Authorized:

Andreas Saldivar, Laboratory Director

Approved & Authorized:

Joseph Coco, QA Manager

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 201 MCE Filter Preparation (DMF Procedure)

Sign: _____ Date: 6/22/10

Print Name: Michael Creighton

Sign: Chon Simpha Date: 6-22-10

Print Name: CHON SIMPHA

Sign: _____ Date: 6/23/10

Print Name: GECarney

Sign: _____ Date: 6/24/10

Print Name: Andreas Saldivar

Sign: _____ Date: 6/24/10

Print Name: Ang Cao

Sign: _____ Date: 11/8/12

Print Name: Bonnie Vanhoff

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 201 MCE Filter Preparation (DMF Procedure)

Sign: _____ Date: Oct 30, 2012

Print Name: ROBERT PRIVETTE
Bob

Sign: _____ Date: 10/30/12

Print Name: KRISHA ORDANIEL
Krisha

Sign: _____ Date: 10/30/12

Print Name: Michael Greenberg

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

It is most important to ensure that contamination of TEM specimens by extraneous asbestos fibers is minimized during preparation.

Upon receipt at the analytical laboratory and before they are taken into the clean facility or laminar flow hood, the sample cassettes are cleaned of any contamination adhering to the outside surfaces with fiber free water.

COLLAPSING SOLUTION

To prepare collapsing solution add 50ml of filter deionized water to a clean graduated cylinder.

Using a disposable pipette add 15ml of glacial acetic acid.

Using a second disposable pipette add 35ml of dimethylformamide (DMF).

Pore the solution in to the DMF jar and mix the solution using a glass stirring rod.

Label the jar with the preparation date and the expiration date (6 months from the preparation date).

FILTER COLLAPSE

Prior to introduction into a class 100 laminar flow hood, cassettes are wet-wiped using fiber-free water.

Care must be taken to ensure that the sampling filter has been separated from the backing filter, if present.

The filter is removed from the cassette using forceps, then cut in half with cleaned surgical scissors.

Check the expiration date of the collapsing solution. If the solution has expired pore any remaining solution into a beaker and place it in the fume hood and prepare new collapsing solution.

Using a micropipette, a 50 ul drop of dimethylformamide (DMF) collapsing fluid* is placed on a cleaned glass microslide; one section of the filter is placed horizontally on top of the solution and allowed to saturate.

Excess collapsing solution is blotted with a paper towel, and the slide is labeled with appropriate identification.

Up to 4 sample sections may be placed onto one microslide. Each slide includes an AMA laboratory blank filter that is assigned a unique number, and accompanies the sample filters through the preparation steps.

Slides containing the filters are transferred in a closed Petri dish to the fumehood. The slides are then placed onto a hotplate at ca. 70 degrees C. for a minimum of 10 minutes.

Samples are plasma etched and carbon coated in accordance with the Standard Operating Procedures (Nos. 204 and 203, respectively).

FILTER DISSOLUTION

Carbon coated filters are transferred to a fume hood for final dissolution of the filter material. Indexed, measured, (refer to SOP Nos. 401 or 402 for Procedure for Measuring Grid Openings) 200 mesh copper grids are placed on a stainless steel mesh resting on a DMF-soaked Kimwipe in a Petri dish.

The DMF level is adjusted so that the mesh of the stainless steel screen is just saturated.

The grids are examined to ensure that the numbering system is not inverted when viewed under the TEM, then are placed directly on the mesh in columns of 3.

The mesh has a small notch cut into one end to indicate the location of the first column of grids. Small (ca. $1mm^2$ x $1mm^2$) filter sections are cut with a cleaned scalpel and placed, carbon side up, onto the grids. Utensils are wet-wiped between each filter.

The top of the Petri dish is labeled with tape to include the sample number(s), grid opening size, time & date, and repairer's initials and placed onto the dish bottom.

Grids are left on the DMF for a minimum of 1 hour. The mesh and grid arrangement is then transferred to a Petri dish containing an acetone-saturated Kimwipe. The preparation is allowed to remain on the acetone for a minimum of 10 minutes to ensure the complete dissolution of the filter material.

Grids are transferred from the mesh to their grid storage box. The contents of the grid storage box are recorded in the Grid Locator log book.

*DMF Collapsing fluid (by volume):
35% DMF 15% Glacial Acetic Acid 50% fiber free water

END SOP 201

Approved & Authorized: _____

G. Edward Carney, Technical Manager

Approved & Authorized: _____

Andreas Saldivar, Laboratory Director

Approved & Authorized: _____

Joseph Coco, QA Manager

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 107 Control and Tracking of Laboratory Consumables

Sign: _____ Date: 6/24/10

Print Name: _____

Sign: _____ Date: 6/23/10

Print Name: GE Carney

Sign: _____ Date: 6/24/10

Print Name: Andreas Saldivar

Sign: _____ Date: 6/24/10

Print Name: Nida McGarvey

Sign: _____ Date: 6/24/10

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 107 Control and Tracking Lab Consumables

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Each consumable and reagent received in the laboratory must be assigned a tracking number, and details of the consumable/reagent must be recorded along with said number in the Laboratory Consumables/Reagent Receipt Log.

- Recorded in the log will be: Product type/name. Supplier. Part # and/or Lot #. Quantity (Number/Size). Date Received. Lab ID. Date Opened. QC Check OK. Date Discarded. Expiration Date.

    - The first five are self-explanatory.

    - Lab ID is a number assigned using the following formula: "YYMMDDX" where "YY" is the two digit year code, "MM" is the two digit month code, and "DD" is the two digit day code, of the date of receipt of consumable/reagent. All consumables/reagents received on a specific day will have the same "YYMMDD", however "X" is a unique letter assigned to each consumable/reagent lot, based upon the order in which they are entered into the logbook. The first consumable/reagent received and logged in on October 13, 2004 would thusly receive the tracking number "041013A". The second consumable/reagent logged in the same day would receive "041013B", followed by "041013C" and so on until the twenty-sixth received tracking number "041013Z". The twenty-seventh consumable/reagent logged in that same October 13, 2004, if such number or in excess of are received, would receive "041013AA" and the twenty-eighth would receive"041013AB".

    - Record the date when the product is first opened. Perform the QC check as specified in the form "Quality Control Check Procedures for Consumables/Reagents". If the QC check is OK, check off as OK and place the record of the QC check in the back section of the logbook. If the QC check fails, retest, and report the problem to the Supply officer if the QC check still fails.

    - Expiration Dates are assigned as follows:

      Primary Standards: one year from date received or expiration date specified by the supplier.

      Reagents: two years from the date received unless a shorter shelf life is specified by the supplier or dictated by the use of the reagent

      Other Consumables (containers and disposable labware): three years from the date received.

      If, when the expiration date is reached a substantial quantity of the product remains, and it appears to be in good condition, the product may be rechecked a new expiration date assigned if the product continues to pass the specified QC checks. The new expiration date shall be a maximum of one year from the date of the last QC check.

  Record the date when the product is disposed of or completely consumed.

END OF SOP 107

Approved & Authorized: _____

G. Edward Carney, Technical Manager

Approved & Authorized: _____

Andreas Saldivar, Laboratory Director

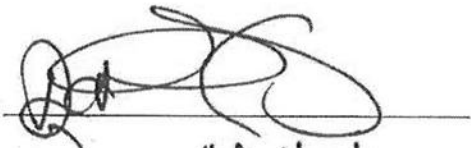
Approved & Authorized: _____

Joseph Coco, QA Manager

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 102 Plan to Transfer Customer Records for Lead Analyses Performed under NLLAP

Sign:  Date: 10/30/2012

Print Name: Dana W Hodson

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 102 Plan to Transfer Customer Records for Lead Analyses Performed under NLLAP

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

In the event that AMA Analytical Services, Inc. ceases operations for whatever business reasons, the following procedure will be used to transfer customer records for lead analyses performed under NLLAP according to the Client's instructions (regarding the handling of in-house samples and records delivery by electronic format or U.S. Mail) and applicable regulations in force at that time. Since customer records are generated with each sample set received for analysis, only customer records associated with in-house sample sets meet the requirement for transfer, the disposition of which is discussed below.

1. Each customer with lead samples in-house will be notified in writing regarding the effective date of cessation of operations, and the procedure regarding pending sample analyses and records for transfer.

2. For analyses just completed, but not yet reported and billed, each customer will be provided with a copy of Client-provided paperwork that was submitted with the sample set, the laboratory report and supporting documentation, and the associated invoice for that sample set when the final report and invoice are completed before the effective date of cessation of operations.

3. For samples received, but not yet analyzed, each customer will have their samples and paperwork returned if the due date for the sample set is after the effective date of cessation of operations.

4. For samples received, but not yet analyzed, AMA Analytical Services, Inc. will analyze the sample set if the due date is before the effective date of cessation of operations, and if the report and invoice can be generated by then. A copy of Client-provided paperwork that was submitted with the sample set, the laboratory report and supporting documentation, and the associated invoice for that sample set will be provided when the final report and invoice are completed.

End SOP 102

Approved & Authorized: _____
Dana Nicodemus Hudson, Director of Client Services

Approved & Authorized: _____
Andreas Saldivar, Laboratory Director

Approved & Authorized: _____
Joseph A. Cook, QA Manager

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 101 Sample Receipt and Log-in Procedures

Sign: _____ Date: 10/30/12

Print Name: Nicole Maxwell
Nicole

Sign: Christine H. Nicodemus Date: 10/30/12

Print Name: Christine H. Nicodemus
Chris

Sign: _____ Date: 10/30/2012

Print Name: Dana Anderson
Dana

Sign: _____ Date: 10/31/12

Print Name: KRISHA OBANNEL
Krisha

Sign: Kim Shipe Date: 11/4/12

Print Name: Kim Shipe
Kim

Sign: _____ Date: 10/31/12

Print Name: Michael Cox
Michael C.

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 101 Sample Receipt and Log-in Procedures

Sign: Michael Greenberg Date: 10/31/12

Print Name: Michael Greenberg
Michael G.

Sign: [signature] Date: 10/31/12

Print Name: Ang Cao
Ang

Sign: [signature] Date: 10/31/12

Print Name: ROBERT PRIVETTE
Bob

Sign: Suphin Chimpad Date: 11/2/12

Print Name: Suphin Chimmapad

Sign: [signature] Date: 11/11/12

Print Name: GEdward Carney
Ed

Sign: [signature] Date: 11/13-12

Print Name: Sygrat Watson

Sign: [signature] Date: 11/13/12

Print: Nida MacGarvey

Signing in Samples

General guidance regarding sample receipt can be found in section 5.1 of this Manual.

Much of the work that we do comes in via FedEx and/or UPS, usually between 0930 and 1030. Open each package one at a time and for each COC in the package, go through the following steps:

- Sign the bottom of the Chain of Custody (COC). Note date and time samples are received by the laboratory and document how samples are shipped to the laboratory (i.e., hand delivered, courier, mail, Federal Express) and condition of the package if damaged. Also note the FedEx or UPS tracking number if appropriate.
- Make sure all forms, especially the COC, are filled out correctly and all necessary information is given.
    - The submittal date, client name and address, contact and submitting persons, job name/location, job and PO #'s, phone and fax numbers should be filled out under the Mailing Address section.
    - The Date & Time Results Required ***must*** be filled out. If this information is not provided or if the information is conflicting, you must contact the client for clarification and note the details of your conversation on the COC. Sometimes the client cannot always be reached for immediate clarification. If so, you should leave a message that the client contact you ASAP regarding the TAT and set the samples aside. If at the end of the day you have still not heard back from the client, do the following:
        1) If no TAT and/or due date has been provided, process and schedule the samples on a standard 5-Day TAT. You must leave a message for the client that this is what you are doing. Be specific, making sure to provide the actual date and time you have scheduled. Note the details of your message on the COC. Any changes and/or additions you make to the COC must be initialed and dated by you.
        2) If the date requested does not match the TAT requested, change the TAT to match the date. Any changes and/or additions you make to the COC must be initialed and dated by you. You must leave a message for the client that this is what you are doing. Be specific, making sure to provide the actual date and time you have scheduled as well as the revised TAT. Note the details of your message on the COC.
        3) We do this so that there is minimal delay in delivering the samples to the appropriate laboratory.
        4) If the client calls back and would like you to schedule samples differently, make those changes immediately and note the details of your conversation on the COC.
    - The following are some special notes on Turn-Around-Time (TAT):
        1) TAT begins when the samples arrive at the lab, and does NOT include weekends.
        2) Immediate/Same-Day: Sample received before Noon are due by 1700-1900 that same day. Samples received after Noon are taken on a case by case basis. We do our best to provide results by 1900 that same day, but you will need to check with the department manager before doing so. In the event that we cannot provide results by 1900 that same day, analysis is scheduled for 1000-1200 the following business day.
            a. Samples that arrive after 1500, which require results the same day should be pre-scheduled and will be billed at the After-Hours rate. Check with a manager before signing in any such samples.
            b. We have a 4-hour TAT for TEM AHERA samples only. If a client is requesting this service you must check with a manager before signing in any such samples. You must also get firm confirmation from a manager at the client's organization that they are willing to pay the expedited rate.
        3) Next Day: Samples are due by 1700 on the following business day. Sometimes a client will request that results be reported by noon on that day. We make every attempt to accommodate this request.

4) 2-Day: Samples are due by 1700 on the 2nd business day. Sometimes a client will request that results be reported by noon on that day. We make every attempt to accommodate this request.
5) 3-Day: Samples are due by 1700 on the 3rd business day. Sometimes a client will request that results be reported by noon at that day. We make every attempt to accommodate this request.
6) 5-Day +: Samples are due by 1700 on the 5th business day (= 7 calendar days). Sample sets with very large volumes(100+ samples depending on the sample type) or are of difficult matrix and/or analytical method may require an additional days to complete. Check with the department manager/supervisor to determine an appropriate date.
7) For samples signed in at 0800, the following TATs apply:
    a. Next Day – samples are due by noon the following business day.
    b. 2-Day – samples are due by noon on the 2nd business day.
    c. 3-Day – samples are due by noon on the 3rd business day.
    d. 3-Day – samples are due by noon three business days later.

- The Sample Data and Sample Analysis Information sections of the COC must also be filled out unless the client has included their field data sheets. If information regarding the analytical method to be used is not available, you must contact the client for clarification and record the details of your conversation with the client. If the client contact be reached, leave a message that the samples will be placed on hold and not processed until such time as the client is able to provide the information necessary. Be sure to record the details of your message on the COC.

- Next, you must inspect the samples to make sure that they have been received in good condition. Sample(s) or sample set(s) shall be voided/rejected at the time of receipt or log-in under the following conditions:

<u>Air Samples & Spore Traps</u>

- If sample cassette has opened during transit.
- The entire sample set is rejected if bulk and air samples are shipped in the same container without being sealed in separate packages.
- Evidence of gross contamination of cassettes or inside of containers.
- The following is a list acceptable sample containers:
    - Lead: 37mm MCE cassettes
    - Asbestos
        - PCM: 25mm, 0.8 or 0.45um cassettes
        - TEM: 25mm, 0.45um cassettes (0.8um cassettes are acceptable under certain circumstances)
    - Mold Non-Viable
        - Spore Traps (such as Air-O-Cell or Allergenco)
        - Collapsed MCE filters microscope slides
        - Greased slides
    - Mold Viable
        - Agar Plates

<u>Bulk Samples (Soil/Solids, Paint Chips, PLMs, NOBs)</u>

- Individual sample to be voided if sample container has ruptured.
- Entire sample set is to be voided if there is evidence of more than one improperly sealed sample container and visible evidence of debris on outside of individual sample container.
- If multiple samples are provided in the same sealed container rather than individually sealed.
- The following is a list of acceptable containers:
    - We prefer that asbestos bulk samples especially be submitted in transparent, sealed containers.
    - Metals: Plastic or Glass containers (zip-lock bags, centrifuge tubes, 4oz jars)
    - Asbestos: Transparent, sealed container (i.e. zip-lock bags, centrifuge tubes, 4oz jars)
    - Mold: Sealed container (i.e. zip-lock bags, centrifuge tubes, 4oz jars)

Water Samples

- Sample container is ruptured
  - Evidence of gross contamination of sample is obvious
  - Improper sample container. The following is a list of acceptable containers:
    - Metals: Plastic or Glass 250mL, 500 mL, or 1 L bottles (6 month holding period)
    - Asbestos: Plastic or Glass 1 L bottles, received chilled at 4 degrees C (48hr holding period)
  - Method specified holding times are exceeded. If TEM waters exceed the holding time, check with a manager, as it is possible to still analyze the sample if certain preparation procedures are followed.
  - Insufficient sample submitted to perform requested analysis

Dust Samples

- Sample container is ruptured
  - Evidence of gross contamination of sample is obvious
  - Improper sample container. The following is a list of acceptable containers:
    - Lead: ASTM Certified wipe in a 50mL Centrifuge Tubes or zip-lock bag
    - Asbestos
      - D5755: 25mm, 0.45um filter cassette
      - D6480: any acceptable lead wipe in a zip-lock bag or other transparent, sealed container
      - Qualitative Dust: same as D5755 and D6480

Vermiculite

- Sample container is ruptured
  - Evidence of gross contamination of sample is obvious
  - Minimum 1oz (preferably 1 gallon) of material in a transparent, sealed container.

If all samples meet these requirements, check the box on the COC that indicates samples were received in good condition. If the version of the COC the samples are submitted under does not have a box, stamp the COC. ~~The following is a list of acceptable sample containers for various matrices:~~

- ~~Next,~~ Finally, it is important to ensure that the requested sample analysis falls within our accreditation status:
- NVLAP covers bulk asbestos by PLM & airborne asbestos by TEM
- AIHA covers airborne asbestos by PCM; lead dust wipes, paint chips, soil/solids, airs; mold spore traps
- NELAC thru NY ELAP covers Asbestos Potable Water by TEM 100.2 & Pb & Cu Potable Water
- We also carry several state specific licenses/accreditations:
  - NY ELAP covers bulk asbestos samples by PLM & TEM for samples originating from NY
  - NY ELAP covers Pb Paint Chips & Dust Wipes for samples originating from NY
    - We are not currently accredited for total Pb analysis on soil/solid materials from NY – these samples may not be accepted.
    - We are not currently accredited for Pb analysis on air samples from NY – these samples may not be accepted **UNLESS** the client confirms in writing that a) the samples are for OSHA personal monitoring **and** b) it is acceptable to them for AMA to analyze the samples outside of our NY ELAP accreditation.
    - We are not currently accredited for PCM analysis on air samples from NY – these samples may not be accepted **UNLESS** the client confirms in writing that a) the samples are for OSHA personal monitoring **and** b) it is acceptable to them for AMA to analyze the samples outside of our NY ELAP accreditation.
  - MD Drinking Water covers asbestos, Pb, & Cu potable samples originating from MD.
  - VA Drinking Water covers asbestos, Pb, & Cu potable samples originating from VA.
  - PR Drinking Water covers asbestos potable samples originating from PR.
  - OH Pb covers lead dust wipes, paint chips, soil/solids, & air samples originating from OH.
  - MA Asbestos covers all bulk and airborne asbestos samples originating from MA.
  - VT Asbestos covers all bulk and airborne asbestos samples originating from VT.
  - VT Pb covers lead dust wipes, paint chips, soil/solids, & air samples originating from VT.

- VA Asbestos covers all bulk and airborne samples originating from VA.
- WV Asbestos covers all bulk and airborne samples originating from WV.
- If samples are received from states and/or for analyses that are not covered by AMA accreditations, check with the Client Services Director, Lab Director, or Lab Manager before accepting.

- Next, make a folder that is labeled with the COC # and the client name. If you are signing in one of our electronic COCs that does not have COC number, assign one by placing a COC # sticker in the upper, right-hand corner of the form. Put the COC and samples in the folder, and the folder in the bin under the appropriate TAT. The bins have been labeled by TAT. Note that this step is necessary only if the volume of samples received warrants it. If the sample volume can be dealt with in a timely manner, than you may skip this step.
- We pay for all sample shipping (except Pb and Asbestos water samples), as long as each shipment of samples amounts to at least $100 worth of analysis. So, you may also want to make a note to yourself which samples were shipped our dime so that as you log them in, you will know what sample sets will need to have shipping charges billed back.

Logging In

- Click on the *Chain of Custody* box.
- Choose the *Enter New COC* box.
- Fill in the information this screen asks for. You should find everything you need on the COC. The 4th line asks for a client code; to find the appropriate client code, click on the arrow at the end of the line and scroll through the clients until you find the one you are working on. You will notice that some clients have more than one code. Usually this is because some clients have more than one office location; make sure you choose the correct location as indicated by the COC.
  - We have a fairly large list of clients who are COD only. This means that you cannot process samples without first arranging for payment. If when you enter a COC into the database, a pop-box comes up saying the client is COD, you must make sure that payment has been secured before you proceed any further with that particular set of samples. Another good indicator that a client may be COD is if they are not already in the database. If you find yourself in this situation, please see a manager for additional instructions on how to handle the sample set.
- Once you have entered all of the information this screen asks for, click on the *Log in Samples* box.
- Use the COC or field data sheets to fill in the information this screen asks for. If you are ever unsure of what code to use, remember that you can click on the arrow at the end of a line to scroll through a list of possible choices.
  - Before you login a single sample, you must confirm that all samples match what is listed on the client submitted paperwork. If there are any discrepancies (i.e. sample numbers do not match, a sample is missing, or sample layers are not clearly identified), contact the client for clarification and record the details of your conversation on the COC. If the client is not immediately available for clarification, leave a message with specific details and record the details of your message on the COC. If only a few samples in the set are in question, you can set those put those samples on hold and continue to process the remaining samples in the set so as to minimize the delay in delivering samples to the laboratory for analysis. Make sure that you notify the client that those samples will be placed on hold until they contact you and note that message on the COC.
- Once you have entered the information for the first sample, click on the *New Sample* box. All information that was just entered will be brought forward except for the "Client Sample #" line. Enter the next sample # and click on the *New Sample* box again.
- Repeat these steps for all samples on the COC. Remember, if more than one type of sample is on the COC, you must change the Analysis Type and TAT lines as necessary to reflect the change in type and price.
- Once you have entered all of the samples on the COC, click on the *Preview Login* to check your work. If everything is correct, print the Login Sheet. If there are any corrections that need to be made, you will need to click on the *Edit or Add to Record* box before any changes can be made.

- Logging in assigns AMA # to every sample that we analyze. Use the Login Sheet as a guide to placing labels with AMA #'s onto the corresponding samples.
- Repeat this process for every COC that you log in.
- Here is a helpful hint to remember; hitting *Ctrl '(apostrophe)* will bring up the information you entered on any given line in the previous record. This is especially helpful when logging in samples that have a long prefixes.
- Once the sample set has been completely logged in and you are certain that all information that appears on the COC and login sheet is correct, deliver that sample set to the appropriate laboratory storage area.

Sample Scheduling

- When you login samples, you enter the date and time due on the Chain of Custody screen. This information automatically appears in the sampling scheduling section of the database.
- When you are processing Same-Day samples, make sure that you notify the appropriate laboratory ASAP, sometimes even before you login the samples.
- You must also be able to schedule the log-in of all samples such that they are delivered to the appropriate department in enough time that all client deadlines can be met. Usually, this means logging in the Same-Day TAT samples sets first, followed by the Next Day sample sets, and so on. However, there are exceptions to this rule. If you are uncertain as to what sample sets should be given login priority, please check with a manager.
- Sometimes certain departments within the laboratory get very busy. When this happens, you will need to keep the lines of communication open with that department manager regarding sample scheduling. The manager may give you a maximum number of daily that samples that you can accept and it will be your responsibility to ensure that you do not schedule more samples than the laboratory can reasonable handle.
    - If we receive more samples than a laboratory can handle, notify the Director of Client Services immediately so that laboratory management staff can begin to work out solutions acceptable to the clients.

End SOP 101

Approved & Authorized: _____

G. Edward Carney, Technical Manager

Approved & Authorized: _____

Andreas Saldivar, Laboratory Director

Approved & Authorized: _____

Joseph Coco, QA Manager

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 410
TITLE: Obtaining a Calibrated Objective Aperture

Sign: _____ Date: 6/23/11

Print Name: Michael Gagel

Sign: _____ Date: 6/23/11

Print Name: GE Carney

Sign: _____ Date: 6/24/10

Print Name: Andreas Saldivar

Sign: _____ Date: 6/24/10

Print Name: Ang Cau

Sign: _____ Date: _____

Print Name: _____

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 410 Obtaining a calibrated objective aperture

Sign: Michael Greenberg Date: 10/30/12

Print Name: Michael Greenberg

Sign: Robert Privette Date: 10/30/12

Print Name: ROBERT PRIVETTE

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 410 Obtaining a calibrated objective aperture

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

This procedure needs to be performed in conjunction with SOP 403, "Procedure for Obtaining a Camera Constant".

When taking the picture that is to be used to determine the camera constant add the following steps.

After the SAED gold ring pattern has been established on the screen and the scope is ready to photograph the pattern insert objective aperture 3 on scope 1 or aperture 2 on scope two. ***See SOP 403 for a detailed description of the set up.***

Using the objective aperture adjustments move the aperture away from the center to the area just outside the first ring.

Start the first 45-second exposure.

At the end of the first exposure depress the film advance button to hold the film plate in place.

Remove the objective aperture and expose the plate for another 45 seconds.

Depress the film advance button to hold the plate for the third exposure.

Remove the beam stop and expose the film again.

Measuring the Objective Aperture

Equation #1
After the film is developed measure the diameter of the aperture image at least 2 times using the loop and the light box and calculate the average.

Equation #2
Divide the camera constant determined from the same negative by 5.3 angstroms to determine the ideal width of one amphibole row spacing.

Divide the diameter of the aperture as measure on the negative by the width of one amphibole row spacing. This is the ideal number of 5.3 angstrom rows that will appear within the aperture when checking a diffraction pattern

A= Camera constant (mm angstroms)
X= Width of one 5.3 angstrom row spacing (mm)
B= Average diameter of objective aperture measured on the negative (mm)
C= Number of 5.3 angstrom rows visible in aperture

Equation #1
X=A / 5.3 angstroms

Equation #2
C=B/X

END SOP 410

Approved & Authorized:

G. Edward Carney, Technical Manager

Approved & Authorized:

Andreas Saldivar, Laboratory Director

Approved & Authorized:

Joseph Coco, QA Manager

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 507
TITLE: Preparation and Analysis of NIOSH 7402 Sample

Sign: _____ Date: 6/23/10

Print Name: GE Carney

Sign: _____ Date: 6-22-10

Print Name: CHON SIMPHA

Sign: _____ Date: 6/22/10

Print Name: _____

Sign: _____ Date: 6/24/10

Print Name: Ang Cao

Sign: _____ Date: 6/25/10

Print Name: Andrew Schalow

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 507 Preparation and Analysis of NIOSH 7402 Samples

Sign: _____ Date: 10/30/12

Print Name: ROBERT PRIVETTE
Bob

Sign: _____ Date: 10/30/12

Print Name: KRISHA GRONNER
Krisha

Sign: _____ Date: 10/30/12

Print Name: Michael Greenberg
Mike G.

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Acknowledgement of "Read & Understood"

By signing this document, you acknowledge that you have read and understood this procedure, and you agree to follow all procedures described in this document.

SOP No. 507 Preparation and Analysis of NIOSH 7402 Samples

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

Sign: _____ Date: _____

Print Name: _____

The purpose of this SOP is to outline the steps to follow for preparation and analysis of NIOSH 7402

Preparation

For MCE filters follow the same procedures outlined in SOP 201 with the exception of the plasma etching steps. Do not plasma etch NIOSH 7402 samples.

For PC filters follow the same procedures outlined in SOP 202.

Evaluation of the Grid

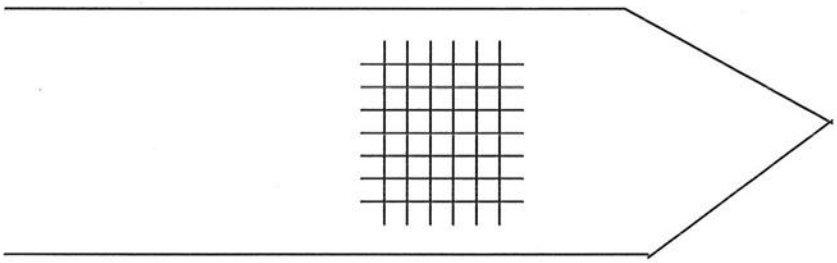
There must be 75% of the carbon replica intact and unbroken for the grid to be acceptable.

Overloading: The method allows for greater than 50% particulate loading across the entire grid before overloading the sample. The restriction on individual grid openings is 20%. For example, a sample with 40% particulate loading may or may not be overloaded. If the sample has large particulate that clumps some of the grid openings may be 50, 60+ percent covered with particulate while others are only 5-10%. In this situation pick 40 grid openings with less that 20% particulate over all 3 grids. If there are not 40 with less than 20% the sample is overloaded. If the particulate was small and evenly distributed there most likely won't be any grid openings with less than 20% particulate.

Analysis

At a magnification of 1,900x, analyze 40 grid openings, regardless of the grid opening size, spread as evenly as possible over 3 grids.

Put the grids in the specimen holder with the grid bars running perpendicular and parallel to the holder as illustrated below. This will allow you to scan a row of grid openings without dropping to low mag.



Starting at the first intact grid opening in a row, analyze all the intact grid openings in the row.

Count all fibers longer than 5 microns with width greater than 0.25 microns that have an aspect ratio of ≥ 3. Count fibers protruding from the grid bar as ½ fibers.

When you encounter a countable fiber, increase the magnification to 19,000x and check the diffraction pattern. For chrysotile, a positive diffraction pattern is sufficient for identification. If the pattern is an amphibole pattern you need to acquire chemical data to positively identify the fiber.

Start another row of grid openings on the first grid. Analyze until you have 13-15 total grid openings on the first grid.

Repeat the procedure for the second and third grids.

Stopping Rules

Analyze until you have completed 40 grid openings or have counted 100 fibers with a minimum of 6 grid openings. You must complete the grid opening that contains the 100th fiber. You cannot stop in the middle of a grid opening.

Calculations and Reporting

The proper way to report a NIOSH 7402 sample is fraction of fibers that are asbestos. Use the following equation to calculate it:

$(f_s-f_b) / (F_s-F_b)$

f_s = Number of asbestos fibers on sample
f_b = Number of asbestos fibers on blank
F_s = Number of Fibers, of any type, on sample
F_b = Number of Fibers, of any type, on blank

You will find that all customers will want the results for the asbestos fibers reported in f/cc and f/mm^2.

For f/mm^2 use:

Number of Asbestos Fibers (s) / Area Analyzed (mm^2)

For f/cc use:

$$\frac{\text{Effective Filter Area (mm}^2\text{)* Number of Asbestos (s)}}{\text{(Area Analyzed (mm}^2\text{)* Volume (L)* 1000 cc/L)}}$$

Quality Control

Follow the procedures outlined in section 13.0 of the Technical Guidelines Manual.

END SOP 507